VERTAC
INCORPORATED Suite 2900—Clark Tower • 5100 Poplar Avenue • Memphis, Tennessee 38137 • 901-761-0050

April 4, 1975

Mr. Don Faggart, Manager
Crop Chemicals Formulation
Mobil Chemical Company
P. O. Box 26683
Richmond, Virginia 23261

Dear Mr. Faggart:

Confirming our telephone conversation, we have completed the engineering work and are prepared to make a preliminary economic proposal for the nitration of the methyl meta chlorobenzoate.

Per our discussions and my letter of March, we feel that we have most of the equipment on hand to start nitration of your raw material in July. The sizing of the equipment is outlined on the attached flow sheet. I have noted those items that would need to be purchased and as we discussed, there would be no problem on delivery with the possible exception of the vacuum source. This equipment, when scaled down from your own batch size of 2200 gallons will give us approximately 300,000 pounds per month production with a 80% operating factor. We feel that with some operating experience, we will be able to possibly shorten the batch time as well as increase our operating factor to supply the product in amounts of 350 to 400,000 pounds per month. If after operating the equipment for a few months, we feel that this is not possible, we can install an additional 1500 gallon vessel and perform the washes and separations outside the reactor. This would increase the number of batches from two to probably three or four per day and would be more than adequate to supply your higher volume.

The cost has been computed on four different production rate ranges. The higher cost for the lower rates assume that we are unable to put other products in the unit during the 10 to 15 days slack time each month. If we are able to put additonal products through the unit during the slack time, then we could adjust these production costs downward. In an effort to share some of this uncovered overhead, I have adjusted the tolling charge to absorb some of the inefficiencies at the lower rate. The cost for the different rates are as follows:

Charges	150-199M lbs.	200-249M lbs.	250-299M lbs.	300M lbs.+
Manufacturing Cost	24¢/lb	18¢/lb.	13¢/lb.	10¢/lb.
Tolling Charge	20¢/lb.	22¢/lb.	25¢/lb.	25¢/lb.
Total Charge	44¢/lb.	40¢/lb.	38¢/lb.	35¢/lb.

Don, as we discussed, this is a tolling arrangement whereby you supply all raw materials in tank car quantities with our supplying a storage tank for the nitric-sulfuric mix as well as the ethylene dichloride. You would also supply a tank car to ship the finished product in so that we would not need a finish product storage tank other than the 2500 gallon surge tank on the flow sheet. Also not covered is any effluent treatment costs. As we discussed, once the treatment specifics have been pinned down, we will treat them in our own facilities if possible at actual out of pocket cost.

Thanks for the opportunity to quote on this project, and we are looking forward to a quick, favorable response. Once you give us the go ahead, we are two to three months away from shipping product. Let us hear from you soon.

Sincerely,

Larry Graybill
Manager
Business Development

LLG:cj

MOBIL NITRATION

BY John Miles & Larry Graybill
April 2, 1975

INDEX

CAPITAL REQUIREMENTS

A.	Purchased Equipment (56,600 in plant, 46,200 to be purchased)	$ 102,800
B.	Installation	47,000
C.	Instruments (Installed)	10,000
D.	Piping (Installed)	60,000
E.	Electrical (Installed)	10,000
F.	Construction Expenses - Misc.	10,000
	TOTAL CAPITAL	$239,800
	CONTINGENCY	35,200
		$275,000

PRODUCTION CAPACITY

8850 lbs./batch (2200 Gal. Reactor)
.682
6036 lbs./batch (1500 Gal. Reactor)
2 batches/Day
12072
.8 Operating Factor
9658
30
289,728 lbs./month

OPERATING COST PER 6036 BATCH

A. Operating Costs/batch

Electrical - 100 HP x 4 hr./batch = 400 hphr x 2.2¢/KWH	$8.80
Steam 8000 lbs./Batch	8.00
Refrigeration 200 tons 50 tons/hr. 15¢/hr/ton	30.00
Cooling H_2O - 5000 Gallons 10¢/M	.50
Operating Labor - 2 m3n/shift	180.00
	$277.30

B. Maintenance Utilities & Labor	36,000.00/yr. (61.65/batch)
Operation & Maintenance Cost	$288.95/batch
	$.048/lb.

C. Capital Depreciation Cost

6 mos. @ 150,000 #/mo. = 900,000
12 mo. @ 350,000 #/mo. = 4,200,000
$275,000 - $5,100.000 # = $.054/lb.

TOTAL PRODUCTION COST $.102/lb.

MAJOR EQUIPMENT REQUIREMENTS

1.	REACTOR		$42,000.00
	1500 Gallon G/L Pfaudler Available 158 ft.^{2} Jacket, Agitator, on Load Cells		
2.	EXTRACTION TANK		
	1500 Gallon 316SS Required w/agitator	Tank	6,200.00
		Agitator	1,500.00
3.	EDC DAY TANK		
	1500 Gallon CS Required		2,000.00
4.	PRODUCT STILL / STRIPPER RECEIVER		3,300.00
		Rework	1,200.00
	2,000 Gallon 304SS Available 7'-0" Dia. x 5'-6 7/8" Straight Side, ASME F & D Heads Rated 140 psi @ 320°F - Good for Full Vacuum		
5.	PRODUCT SURGE TANK		3,500.00
		Rework	1,000.00
	2500 Gallon 347SS Available 7'6" Dia. x 7'6" Jacketed		
6.	STRIPPER COLUMN		
	2' Dia. x 15 1/2' w/1 1/2" Dia. SS Pall Rings	Available	2,600.00
		Rework	1,000.00
7.	REACTION COOLER		12,000.00
	350 ft.^{2} 316SS Required		
8.	STRIPPER HEATER		8,400.00
	200 ft.^{2} SS Required Full Vacuum Rated @300°F		
9.	EDC CONDENSER		2,000.00
	100 ft.^{2} CS Required Full Vacuum Rated @ 300°F Use 2 - 60 ft.^{2} Glass in series available		
	SUB-TOTAL MAJOR EQUIPMENT		$86,700.00

PUMPS

1.	R. M. Charge Pump 20 min. at 24 GPM = N_2	
2.	98% HNO_3 10 min. @ 3 GPM 225 min. @ 0.6 GPM	$1,000.00
3.	98% H_2SO_4 220 min. @ 1.5 GPM	1,000.00
4.	EDC to Reactor 20 min. @ 31 GPM 2 x 1 - 80' N	1,500.00
	EDC to Extraction 20 min. @ 33 GPM	
5.	EDC fr. Extraction to DT 20 min. @ 33 GPM 2 x 1 -80' N	1,500.00
	Drain 85% H_2SO_4 20 Min. @ 17 GPM	
6.	Spent Acid to Extraction 20 min. @ 18 GPM 1 1/2 x 1-60'N	1,500.00
	Drain $(NH_4)_2$ SO_4 20 min. @ 8 GPM	
7.	Reactor Circulation 200 - 250 GPM 3 x 2 - 60' N	2,800.00
	Product to Still 6 min. @ 200 GPM	
8.	Still Pump 150 - 200 GPM 3 x 2 -100'	2,000.00
9.	Product Pump 100 GPM	1,800.00
		$13,100.00
10.	Vacuum System 100 GPM @ 29.5" Hg Vac	3,000.00
	Total Pumps & Vacuum	$16,100.00
TOTAL OF EQUIPMENT, PUMPS, AND VACUUM		$102,800.00

April 2, 1973

ESTIMATED MANUFACTURING COST Prepared by L. Graybill Checked by John Miles

1. Plant Production
   Rate/Day 12,072 (2 batches per day at 6,036 lbs./batch)
   Day/Yr. 292 (80% Operating Factor)
   Total Production Cost/Pound

2. Raw Material & Fuel Cost				Cost/Batch	Cost/lb.
Type	Units	Unit Cost	Usage		
Subtotal Raw Materials & Fuel				$	$

3. Utilities					
Type	Units	Unit Cost	Usage		
Electricity	Batch	8.80			
Steam	Batch	8.00			
Water-Cooling	Batch	.50			
Refrigeration	Batch	30.00			
Subtotal Utilities				$ 47.30	$ .0098
4. Operating Labor No. Operators/shift 2				180.00	.027
5. Rentals					
6. Burden					
A. Repair Labor and Supplies				61.65	
B. Repair Supplies					
C. Operating Supplies				7.15	
D. Supervision 50% (OL + OH)				135.00	
E. Maintenance Allocation				61.65	
Subtotal, Burden				$ 265.45	$ .0440
7. Fixed and Service Costs					
A. General Works Expense incl. taxes & ins. 50,000/yr.				85.62	
B. Depreciation 8 yr.S.L. 275,000=34,375/yr.÷ 584				58.86	
C. Laboratory .25 Tech./Batch				13.00	
D. Interest Expense 275,000-18 mos.@12% ÷ 18 mos.				55.99	
Subtotal Fixed & Service Costs				$ 213.47	$ .0354
8. Total Bulk Cost				706.22	
9. Loading, Packing & Shipping					
A.					
B.					
Subtotal L, P & S				$	$
10. Contingencies 10%				79.62	.0132
11. Total Manufacturing (Batch Basis)				785.84	
12. Unit Manufacturing Cost for production of 6,036 lbs.					.1302

Notes:

Variable cost for monthly production at following rates:

	150-199M lbs.	200-249 M lbs.	250-299 M lbs.	300 M lbs. +
Utilities	.0098	.0098	.0098	.0098
Operating Labor	.0522	.0390	.027	.0195
Burden	.0851	.0638	.044	.0319
Fixed and Service Cost	.0683	.0513	.0354	.0256
Contingency	.0250	.019	.0132	.0099
Total Manufacturing Cost	.2404	.1829	.1294	.0967

VERTAC
INCORPORATED
Suite 3200 — Clark Tower
5100 Poplar Avenue
Memphis, Tennessee 38137

INTER-OFFICE CORRESPONDENCE

April 14, 1975

TO: Ray Guidi, John Miles ✓, Bill Shackelford, Niven Morgan

FROM: Larry Graybill

SUBJECT: Mobil Nitration

REPLY REQUESTED BY (DATE)

Attached is the preliminary engineering work by John Miles on adapting the nitration expansion to Mobil's process.

As you recall we have been discussing the utilization of our Eagle River nitration facilities for the nitration of Mobil's intermediate for their Modown production. They have asked for a preliminary proposal on a 150,000 pounds per month production July through December, 1975; then 350,000 pounds per month for the calendar year, 1976.

Based on this information the letter of quotation which is also attached was mailed to Mobil April 4, 1975. I will be following up with Mobil for their response.

Larry Garybill

LLG/bc

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April 4, 1975

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Sincerely,

Larry Graybill
Manager
Business Development

LLG:cj

9/11/75

MOBIL VISIT

G.F. MATHER
J. WOLFENBARGER-MOBIL CHEMICAL
J. ELLINGTON-MOBIL CHEMICAL

(1) USE CBE T/C AS "NURSE TANK". PROVIDE FOR FEED T/T TO T/C.

(2) CBE SUBCOOLS PROBABLY WON'T FREEZE IN T/C-NORMALLY CLEAR SOMETIMES SHOWS UP PINK. CHECK FOR ACID TO PREVENT BAD CORROSION. CBE QUALITY HAS BEEN POOR LATELY.

(3) OCCASIONALLY CBE T/C RELIEF WILL SHOW UP SET BELOW 25PSIG/REPLACE RV WITH 25PSIG UNIT.

(4) H2O LAYER ON EDC EACH BATCH MUST BE DEALT WITH. SIDE NOZZLES ARE A POSSIBILITY. H2O LAYER IS A CONTINUAL HEADACHE OF THE OPERATION.

(5) ANALYZE EDC/CBE FOR CONCENTRATION AND H2O NORMAL H2O 1%WT/5% WT TOO MUCH WILL KILL RXN.

(6) ADD TI & PI TO TM104 IN CASE H2O IS ADDED WITH EDC.

(7) ALL ALUMINUM VALVES ARE A PROBLEM, SUGGEST ALL STAINLESS VALVES OR ALUMINUM HOUSING/STAINLESS INTERNAL BALL VALVES LOTS OF WELD PROBLEMS WITH ALUMINUM PIPING ALSO.

(8) SUGGEST FLEX COUPLINGS AROUND PUMP AS ALUMINUM PIPING IS WEAK. WHITE FUMES DUE TO POROSITY/LOCATE A WATER HOSE NEAR TANK/PUMP SEALS ALSO A PROBLEM.

(9) STRAINER ON PM101 AND PM102 TO REMOVE ACID RUST, GUNK, ETC.

(10) IN EDC SERVICES GASKETS, ETC. MUST BE TEFLON. NITRIC WILL CHEW UP ORGANIC OR CARBON MATERIALS ALSO.

(11) DESIGN MIXING CHAMBER FOR A VENTURI EFFECT (PINCH NOZZLE SIZE OF BOTH ADDITION POINTS). INSERT ACID PIPING INTO CIRCULATION LOOP.

(12) MINIMIZE ACID INLET TO AM101 TO E-M101 PIPING DIMENSION.

(13) TO MAINTAIN CaCl2 BRINE AND PREVENT CORROSION CHECK PH 8-10, 02 KEEP IT OUT SHOULD NITROGEN PAD BRINE TANK, AND MONITOR CHROMATE.

(14) DON'T RUN @32°F AS ORIGINALLY SUGGESTED. WILL DEVELOPE SEVERE FREEZE PROBLEMS. RUN AT 45°F (42-47°F RANGE).

(15) RUN BRINE AT SAY 10°F
(6-16°F) MOBIL FROZE UP A BATCH AND THEN OVER HEATED.

(16) DID FINISH BATCH BUT WITH YIELD LOSS.

(17) POSSIBLY 15% PREADDITION OF HNO3 WOULD HELP ALLEVIATE FREEZING PROBLEM BUT MOBIL HAS MIXED FEELINGS ABOUT WHETHER OR NOT THIS TECHNIQUE IS REQUIRED.

(18) CHARGE AN EXCESS OF 3-4% HNO3 INSTEAD OF 1% EXCESS HNO3
--H2SO4 CONSUMPTION HAS BEEN REDUCED SUBSTANTIALLY--

MOBIL VISIT

G.F. MATHER
J. WOLFENBARGER - MOBIL CHEMICAL
J. ELLINTON - MOBIL CHEMICAL

(19) SAMPLE R-M101 AT INCREASING FREQUENCY FOR UNREACTED CBE AS THE END POINT IS REACHED. START AT ONCE PER HOUR, THEN ONE PER HALF HOUR, ETC. THIS TECHNIQUE CAN SUBSTANTIALLY DECREASE H2SO4 CONSUMPTION.

(20) ADD TI ON T-M106 AND T-105. SPENT ACID CONCENTRATION NOW IN THE 80 TO 83% H2SO4 RANGE.

(21) AUTO SELECTOR VALVES USED BY MOBIL FOR OPERATING FUNCTIONS.

(22) LIGHT BROWN TO MOTOR OIL COLOR ON ACID PHASE OFF R-M101.
HAZY GREEN/BROWN FOR PRODUCT PHASE FROM R-M101.

(23) .2% CBE IN SPENT ACID
4.8% CBE IN FEED TO TM04

(24) CONSIDER WATTMETER ON P-M110 & M-M101

(25) AMMONIA/H2) CHECK WITH PH PAPER ON THE (8) LOOP. WE WILL GET A CRUD LAYER (MIXTURE OF NH4OH, ACID, EDC AND NBE) FROM THE NEUTRALIZATION WASH.

(26) MOBIL STRONGLY SUGGESTS AN INTERFACE TANK TO HANDLE 300-500 GALLONS PER BATCH OF THIS CRUD LAYER. A 1000-2000 GALLON TANK IS DESIRED. ONE WHICH WAS SET UP FOR SECONDARY PHASE SEPARATION AND RECOVERY. EAGLE RIVER'S POSITION IS THAT SUCH A SYSTEM WILL BE ENGINEERED AND CONSTRUCTED AS A "SCOPE CHANGE" ITEM NOT WITHIN THE NOVEMBER 1, STARTUP DATE.

(27) DOUBLE SETTLING TIME ON NH4OH/PRODUCT TO ONE HOUR. BUMP AGITATOR AFTER 1/2 HOUR.

(28) PRODUCT PHASE CHANGES FROM LIGHT BROWN TO DARK PITCH BLACK FOR SOMETIME PRIOR TO THE ORGANIC/AQUEOUS INTERFACE.

(29) AMMONIUM SULFATE IN STRIPPER, IN NBE, IN SPENT ACID IS A PROBLEM. CRYSTALS ARE SAND LIKE AND CAUSE PLUGGING.

(30) AMMONIUM SULFATE CRYSTALS COULD GO AS FINE A 1 MICRON.

(31) PROVIDE DRAIN ON TM106 FOR EDC TAKE OFF

(32) AGITATOR? ON V-M101. MOBIL UNDECIDED AS TO DESIREABILITY.

(33) PROVIDE HAND OPERATED CONTROL VALVE (GLOBE VALVE ETC). ON E-M104 STEAM

(34) 50-60 MM HG @270°F SUFFICIENT FOR V-M101 STRIPPING

(35) INSTALL SHORT DIP PIPE ON V-M101 CIRCULATION RETURN

(36) ACTUAL NBE FREEZE PT WITH SUBCOOLING 90°F.
.5% EDC (.2-.4$ TYPICAL) SPEC.

MOBIL VISIT

G.F. MATHER
J. WOLFENBARGER-MOBIL CHEMICAL
J. ELLINGTON-MOBIL CHEMICAL

(37) CORRECT VOLUMES ON TANKS SHOWN ON P & ID

(38) PRODUCT TANK, STRIPPER, ETC. WILL ACCUMULATE AMMONIUM SULFATE WITH TIME.

(39) 600LB RATING ON NH3 PIPING RECOMMENDED

(40) AQUA NH3 NORMAL RANGE 12% NH3
10-15% NH3

(41) AN EDC LAYER WILL ACCUMULATE IN SUMP AND NEEDS TO BE SETUP FOR RECOVERY/ALSO EFFECT ON PUMP MATERIALS OF CONSTRUCTION

(42) CONSERVATION VENT ON T-M107 SUGGESTED TO PREVENT NH3 LOSS.

(43) JOHN ELLINGTON WILL PROVIDE UPDATED STANDARD OPERATING CONDITIONS TO REFLECT THE LATEST MOBIL RUN.

(44) POSSIBILITY OF WATER WASH IN R-M101 INSTEAD OF AMMONIA WASH.

(45) SUGGEST ONE HOUR FOR AQUEOUS PHASE SEPARATION INSTEAD OF 1/2 HOUR.
ONE HOUR FOR EDC AND CBE ADDITION.
SHORTEN ACID ADDITION TO ONE HOUR.

February 23, 1976

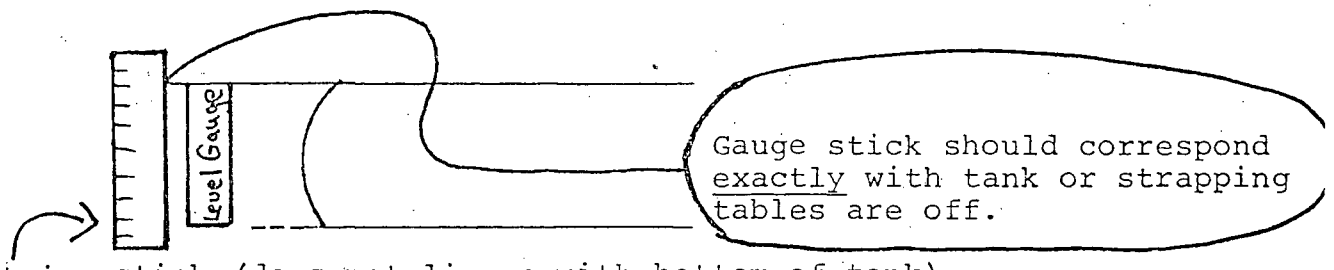
MOBIL TOUR

TO: John Miles/John Holcomb (per George F. Mather/Linnie Duncan)

(1) Effluent line (Plastic portion) is broken enroute to BASALIN tank farm effluent sump. Aqueous effluent is draining toward East Blackhawk Warehouse via their spur.----Suggest all carbon steel line as plastic line was a temporary measure only.------ Also this area along the north property line and railroad tracks needs a good trash pickup and then may be some gravel.

(2) General trash between P-10 cooling water tower and Mobil Tank farm. Area east of Mobil Tank farm. Both areas could use finish gravel that was started around rest of unit.

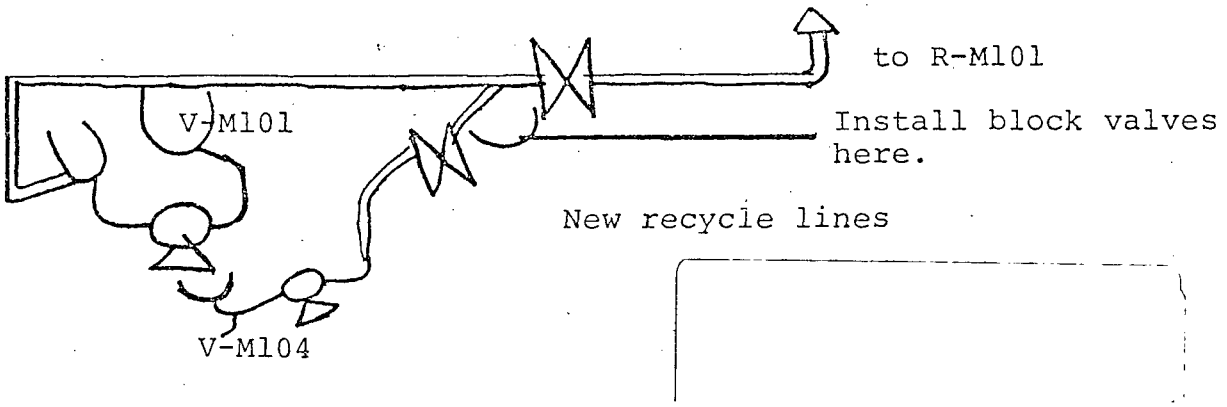
(3) The T-M103 (EDC) Level stick is about 4½" "off" from the Tank. Although a minor point, this oversight hurts in our effort to control EDC losses. Have Robert Wooten's group line-up properly.



ft.in. stick (does not lineup with bottom of tank)

(4) T-M104 level indicator is a menace as it continually plugs with ammonium sulfate (like T-M103 used to). Install dipleg or side mtd btm level tap. Operators should be using T-M103 level gauge for filling T-M104 as it is inherently much more accurate. T-M103 has a better gauge table and has a level glass. T-M104 is a dp cell which measure liquid head. Since T-M104 has materials with different gravities (acid and EDC) it is not that close. Therefore, the T-M104 level indicator is a guide to the operator only.

(5) To make use of the recycle lines installed on the turn around for the stripper and extractor two valves need to be installed:



(6) The brine control on E-M101 is confusing and is hard to control. The ideas are fine but some changes are needed or operators will all be using manual system again. Basically:

(1) The by-pass control valve should be an on or off valve (from panel board)

(2) The brine to the exchanger should be on the air regulator control.

Trying to tie both systems is (a) confusing & unduly complex.

(b) It doesn't work because cooling load changes with perf. of the refrigeration machine, stage in the reactor batch, etc.

(7) Floy's review of MOBIL paper work indicates that the MOBIL operators are doing a fine job on batch logs and plant operating conditions. Several points:

(a) Some dates and analysis are missing on batch sheets

(b) Some extraction batches are noticeably short of EDC charge (standard condition is 1050 gal EDC-----batches as low as 500 gals EDC have been noted)

(8) Operators are storing filter housing in storage room behind control room. Need to store outside in a cabinet, etc. (as product odors don't belong in control room).

12/5/75

Bill Shackelford —

Jim Lynn —

Congressional Record

R

Eagle River Chemical Corp.

Highway 242

WEST HELENA, ARKANSAS 72390

TELEGRAM

December 3, 1975

NDM
JCB cc: 12-4-75 ll
fyi
R

Mr. Don Taggart
Mobil Chemical Company
Richmond, Virginia

As of 0700 12/3/75, we have on hand 47,600 pounds of finished MBE. On 11/28/75 we shipped 35,400 pounds MBE to Mount Pleasant. This total of 83,000 pounds MBE will satisfy the 12/4/75 contract commitment.

JOHN MILES
EAGLE RIVER CHEMICAL

cc: R. Gould
B. Fabian

Tube Project $555-565,000 (Est.)
Will required addition $10-20,000 (items Mobil suggested)
Operation running well - raw materials on hand

R

Well done!!

7/21/75

JERRY:

THE MOBIL NITRATION PROJECT IS GETTING VERY CLOSE TO GO. WE WOULD APPRECIATE YOUR HELP IN SECURING INFORMATION ON KEY EQUIPMENT ITEMS WHILE JIM AND I CARRY THRU WITH THE ENGINEERING. IF YOU COULD LOCATE "CANDIDATE" ITEMS THRU USED EQUIPMENT DEALERS, OTHER VERTAC LOCATIONS, ETC., IT WOULD BE A REAL HELP. SEE ATTACHED FOR DETAILS.

TIMING - THIS WEEK

THANKS,

GEORGE

CC: BILL SHACKELFORD
JOHN HOLCOMB
JIM FOWLER
GEORGE MATHER
JOHN MILES
BOB FABIAN

KEY EQUIPMENT ITEMS MOBIL NITRATION
(IN ORDER OF PRIORITY)

(1) R-M100, NITRATION REACTOR STAINLESS STEEL CONSTRUCTION, NOMINAL 3000-3500 GAL. WORKING VOLUME, CONDITION OF INTERNAL METAL SURFACES SHOULD BE EXCELLENT BECAUSE OF SERVICE INTENDED (NITRATION RXN). WEIGHT OF UNIT IS IMPORTANT.

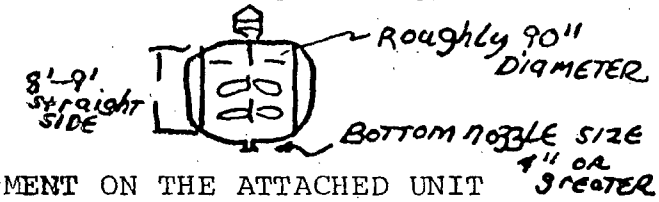
DESIRABLE CHARACTERISTICS IN ROUGH ORDER OF PRIORITY

(A) 15 HP (OR GREATER) AGITATOR DRIVE WITH TURBINE AGITATOR.

(B) JACKETED OR INTERNAL COILS.

(C) 15 PSIG (OR GREATER) VESSEL PRESSURE RATING.

(D) FAIRLY STANDARD DIMENSIONS (ROUGHLY 90" DIAMETER BY 8' to 9' HEIGHT) AS OPPOSED TO ODDBALL SPECIAL CONFIGURATION:



(5) I CALLED AARON EQUIPMENT ON THE ATTACHED UNIT WHICH IS QUITE SUITABLE BUT IT SEEMS EXPENSIVE.

(2) V-M100, SOLVENT STRIPPER, 3000 GAL. WORKING VOLUME, STAINLESS STEEL CONSTRUCTION. FULL VACUUM RATING.

OTHER DESIRABLE CHARACTERISTICS

(A) JACKETED

(B) 15 PSIG (OR GREATER) VESSEL PRESSURE RATING

(C) FAIRLY STANDARD DIMENSIONS

(D) CAPABLE OF HAVING AN AGITATOR ADDED AT A LATER DATE

(3) E-M100, NITRATION REACTOR COOLER, NOMINAL 600 FT^2 SURFACE AREA, STAINLESS TUBES, CARBON STEEL SHELL. CONDITION MUST BE VERY GOOD.

(4) E-M103, STRIPPER HEATER, NOMINAL 400FT^2 SURFACE AREA, STAINLESS TUBES, CARBON STEEL SHELL WITH 150 PSIG RATING.

(5) E-M101 & E-M102, CONDENSERS, 100FT^2 EACH, STAINLESS OR CARBON STEEL CONSTRUCTION.

Phone: (501) 572-3701

P. O. Box 2648

Eagle River Chemical Corp.

Highway 242

WEST HELENA, ARKANSAS 72390

TELEGRAM

December 3, 1975

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Richmond, Virginia

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JOHN MILES
EAGLE RIVER CHEMICAL

cc: R. Guidi
B. Fabian

MOBIL OPERATING DATA

(Run to date thru first 69 Batches)
2-9-76

		AVERAGE	RANGE	REMARKS
I.	TOTAL CYCLE TIME R-M101	17.84 hrs.	10.08-28.17 hrs.	Numerous batches in + 12 hour range prior to acid pump problems.
	a. CBE & EDC Addition Time	1.09 hrs.	.33- 7.58 hrs.	
	b. Total Acid Addition Time	7.45 hrs.	2.08-14.25 hrs.	
	c. ø Separator Time (1st)	1.46 hrs.	1.00- 7.50 hrs.	
	d. Neutralization Time	.76 hrs.	.25- 2.87 hrs.	
	e. ø Separator Time (2nd)	1.61 hrs.	.67-10.00 hrs.	
II.	TOTAL CYCLE TIME T-M104	4.63 hrs.	2.08-18.42 hrs.	
II.	TOTAL CYCLE TIME V-M101	7.81 hrs.	2.33-21.00 hrs.	Often a function c boiler performance

(KSC) → J. MILLS

United States Patent [19]

Theissen

[11] **3,862,209**

[45] **Jan. 21, 1975**

[54] **PROCESS OF MAKING NITRO PHENOXYBENZOIC ACID ESTERS**

[75] Inventor: **Robert James Theissen,** Union County, N.J.

[73] Assignee: **Mobil Oil Corporation,** New York

[22] Filed: **May 12, 1972**

[21] Appl. No.: **252,780**

[52] **U.S. Cl.** ..... **260/471 R**

[51] **Int. Cl.** **C07c 101/54**

[58] **Field of Search** ..... 260/471 R

[56] **References Cited**

UNITED STATES PATENTS

3,013,054 12/1961 Richter 260/501.16

Primary Examiner—Lorraine A. Weinberger
Assistant Examiner—L. A. Thaxton
Attorney, Agent, or Firm—Charles A. Huggett; Howard M. Flournoy

[57] **ABSTRACT**

Improved process for the synthesis of certain nitro phenoxybenzoic acid esters in which high yield, high purity, herbicidally effective compounds are obtained. In general the improvement comprises:

1. esterification of a halobenzoyl halide with isopropanol thereby facilitating separation of isomeric impurities from the desired isopropyl-nitrobenzoate ester intermediate.
2. transesterification of a phenoxy derivative of said ester to the desired phenoxybenzoic acid ester.

9 Claims, No Drawings

PROCESS OF MAKING NITRO PHENOXYBENZOIC ACID ESTERS

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is related to Ser. No. 819,412, filed Apr. 25, 1969 now U.S. Pat. No. 3,652,645, and Ser. Nos. 194,479 now U.S. Pat. No. 3,776,715, 194,480 now abandoned and 194,481 now abandoned, all filed Apr. 16, 1970, and all entitled HALOPHENOXYBENZOIC ACID HERBICIDES and Ser. No. 114,712 now U.S. Pat. No. 3,784,635, filed Nov. 19, 1970 and entitled SUBSTITUTED PHENOXYBENZOIC ACIDS AND DERIVATIVES THEREOF AS HERBICIDES. Ser. Nos. 194,479, 194,480 and 194,481 are divisions of Ser. No. 819,412. Ser. No. 114,712 is a C-I-P of 819,412.

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention is concerned with a novel process of making certain phenoxybenzoic acid compounds which are useful as herbicides.

2. Description of the Prior Art

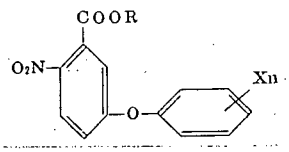
In U.S. Pat. No. 3,013,054, French Pat. No. 1,502,538, and co-pending U.S. Ser. No. 819,412 respectively the use of 2-methoxybenzoic acids, 4-phenoxybenzoic acids, and 5-phenoxybenzoic acids as herbicides is disclosed.

These compounds are readily prepared by the Ullman ether synthesis reaction between, for example an alkali metal salt of a halophenol and a halonitrobenzoic acid. An amide, ester or salt of the benzoic acid may be used, however, the ester is preferred. The halonitrobenzoic acid can be prepared by nitrating a halotoluene and subsequently oxidizing the methyl group, or by subjecting the acid to direct nitration.

However, prior art methods of synthesizing these compounds often resulted in low yields of the desired product or a product containing undesirably high amounts of impurities or both. It is the discovery of this invention that the subject phenoxybenzoic acid esters can be synthesized in high yields with a minimum amount of impurities.

SUMMARY OF THE INVENTION

This invention provides an improved process for making herbicidally effective compounds having the following general structure:



where R is alkyl ($C_1 - C_5$) branched or unbranched, X is a member selected from the group consisting of hydrogen, iodine, fluorine, chlorine and bromine, and *n* is an integer from 2 to 5, and in which compound at least one X is not hydrogen.

More specifically this invention provides a process for synthesizing phenoxybenzoic acid esters having the above general structure comprising:

1. esterifying a halobenzoyl halide with isopropyl alcohol;
2. nitrating the resulting halobenzoate ester;

3*a*. reacting a halophenol with an alkali metal carbonate, bicarbonate or hydroxide; and

3*b*. reacting the resulting alkali metal phenolate with the nitrated ester;

4. transesterifying the resulting halophenoxynitrobenzoate in an excess amount of an appropriate alcohol in the presence of a catalyst selected from the group consisting of alkali metal or alkali metal alkoxide to the desired phenoxybenzoic acid ester.

This invention also provides, in a process for synthesizing phenoxybenzoic acid esters, means for separating undesired isomeric impurities from the desired isopropyl nitrobenzoate ester intermediate comprising esterifying a halobenzoyl halide with isopropyl alcohol thereby facilitating separation of said isomeric impurities from the nitrated isopropyl ester. Thus, this application provides a means of conveniently nitrating the product of a halobenzoyl halide/isopropyl alcohol esterifying reaction, recovering same and then reacting such nitrobenzoate with a suitable alkali metal phenolate to obtain a halophenoxynitrobenzoate and subsequently transesterifying it to the desired halophenoxynitrobenzoate ester. The phenoxy derivative of the nitrated benzoate is accordingly transesterified to the desired halophenoxynitrobenzoic acid ester with an excess of an appropriate alcohol (C_1–C_5) in the presence of a catalyst selected from the group consisting of an alkali metal or alkali metal alkoxide.

DESCRIPTION OF SPECIFIC EMBODIMENTS

This invention, therefore, provides a multi-step process for synthesis in which phenoxybenzoic acid esters are prepared in high yield and high purity. Impurities associated with prior art processes are thus substantially eliminated or minimized within acceptable limits.

Non-limiting examples of compounds conveniently prepared according to this invention are:

methyl 5-(2′,4′-dichlorophenoxy)-2-nitrobenzoate;
methyl 5-(2′,4′,6′-trichlorophenoxy)-2-nitrobenzoate;
propyl 5-(2′,4′,6′-tribromophenoxy)-2-nitrobenzoate;
methyl 5-(2′,3′,4′,5′,6′-pentachlorophenoxy)-2-nitrobenzoate;
n-pentyl 5-(2′,4′,6′-trichlorophenoxy)-2-nitrobenzoate;
methyl 5-(2′-chlorophenoxy)-2-nitrobenzoate;
ethyl 5-(2′-chloro-4′-fluorophenoxy)-2-nitrobenzoate;
methyl 5-(2′,4′-dichloro-6′-fluorophenoxy)-2-nitrobenzoate;
methyl 5-(4′-chloro-2′,6′-dibromophenoxy)-2-nitrobenzoate.

In general the invention comprises (1) esterification of a halobenzoyl halide, (2) nitration of the resulting ester, (3*a*) preparation of a phenol salt of an alkali metal carbonate, bicarbonate or hydroxide and (3*b*) reaction thereof with the nitrated ester and (4) transesterification of the nitrated phenoxy compound to the desired phenoxybenzoic acid ester.

In Step 1 a halobenzoyl halide, e.g., m-chlorobenzoyl chloride, is esterified with isopropanol. Nearly quantitative yields are obtained. The benzoyl halide can be a fluoride, bromide, or an iodide or combinations of two or more halides, such as chlorobenzoyl fluoride. Preferred compounds are chlorobenzoyl chloride and chlorobenzoyl fluoride. Alcohols other than isopropa-

nol can be used. However, isopropanol is especially preferred. Step 2 involves the nitration of the ester. Nitration is conveniently accomplished in concentrated nitric acid (90–100%) with a mole ratio of nitric acid to ester substrate between about 5–20 to 1. The temperature of the nitration reaction can vary from about −50° to about +25° C or higher, e.g. with branched chain alcohols, temperatures of up to about +45° C are suitable. The distribution of isomers produced in the nitration step is believed to be temperature dependent. Generally, a progressively higher percentage of the desired 5-chloro-2-nitro isomer is obtained at lower temperatures.

Even though the desired isomer is the predominant product, separating it from unwanted isomers poses a difficult problem. The primary isomeric impurities are the 3-chloro-2-nitro and the 3-chloro-4-nitro. It is practically impossible to separate them from the desired isomer by distillation or chromatography, and repeated fractional crystallization is tedious and time consuming. However, if isopropyl alcohol is the esterifying agent, obtaining the desired isomer is greatly facilitated. The unique nature of the isopropyl esters is such that the desired isomeric ester (5-chloro-2-nitro) has the highest melting point and the major isomeric impurity (3-chloro-2-nitro) is an oil. Since, additionally, some of the 3-chloro-4-nitro can also be separated by a sustained vacuum filtration technique ultimate product yield and purity are greatly improved.

A typical composition of solids and oils obtained after work up of the isopropyl esters following nitration at −20° C is shown below:

	%Yield	5-Cl-2-NO_2	3-Cl-4-NO_2	3-Cl-2-NO_2
Solid	78.2	96.4	0.9	2.7
Oil	18.1	30.4	9.7	55.9

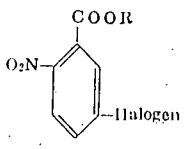
Subsequent recrystallation of the solid yields 98–99% purity of the desired isomer, however, recovery may be diminished because of the ready solubility of the compound in organic solvents. Nonetheless, applicant's discovery of the unique nature of the isopropyl esters significantly increases product purity and product yield.

The phenol salt may be prepared by a variety of known means from an alkali metal carbonate, bicarbonate or hydroxide in an aprotic organic solvent, such as dimethylacetamide, dimethylformamide or dimethylsulfoxide. Preferred alkali metals are Na and K. During the reaction an aromatic cosolvent, e.g. toluene or xylene can advantageously be used to remove water. Any suitable halophenol can be used. However, chlorophenols are preferred.

The alkali metal phenolate so produced is reacted with the nitrated ester produced in Step 2 above. This reaction can take place at temperatures from about 70° to 200° C, but preferably at about 140° to 160° C. The Step 3*b* reaction is accompanied by side reactions and the presence of undesired isomers which affect the purity of the desired product. Consequently the isopropyl ester intermediate is preferred because, as stated above, it minimizes these difficulties.

The purity and yield of the product recovered in Step 3*b* is also affected by various factors, interalia, the structure of the R group, the type of halogen in the intermediate product (isopropyl-5-chloro-2-nitrobenzoate) and the nucleophilicity of the phenol salt.

EXAMPLE OF YIELDS IN STEP 3*b*



1) Dichlorophenol Salt

R	Halogen	Maximum Isolatable Yield
Methyl	Cl	60
Ethyl	Cl	85
Isopropyl	Cl	93

2) Trichlorophenol Salt

R	Halogen	Maximum Isolatable Yield
Methyl	Cl	28
Methyl	Br	35
Ethyl	Cl	45
Isopropyl	Cl	80

It is apparent, therefore, that the best yields with minimum by-products are obtained using the isopropyl ester intermediate.

In the final step, transesterification of the isopropyl ester of Step 3*b* to a desired phenoxybenzoic acid ester is accomplished. The transesterification can conveniently take place in a refluxing solution of an excess amount of the appropriate alcohol catalyzed by an alkali metal or the corresponding alkali metal alkoxide. Preferably the corresponding alkoxide is used. Appropriate alcohols are, e.g. lower alkyl (C_1–C_5) such as methyl or ethyl. Any alkali metal is suitable. Na or K are preferred.

The percentage of conversion to the desired phenoxybenzoic acid ester varies with the molar amount of the alcohol used. A 25:1 molar ratio of alcohol to the isopropyl ester intermediate yields about a 96% conversion. This amount of solvent is also most convenient for isolation of the desired product which precipitates directly from the reaction solution. Yields in the range of 85–90% are readily obtained with product purity of about 99%. The residue from the mother liquor may be reworked to obtain additional product.

The nature of the catalyst is apparently critical since only alkali metal or alkali metal alkoxide catalysts have proven to be very effective for the transesterification. About 3 to 10 molar percent of the catalyst is usually required.

The Step 3*b* product, halophenoxynitrobenzoate ester, can be isolated and recovered. Alternatively immediately after vacuum distillation of the solvent of Step 3*a*, the transesterification alcohol and catalyst can be added directly to the residue and transesterification to the desired phenoxybenzoate ester accordingly carried out. Residual alkali metal chloride from Step 3*b* may be removed from the warm solution by filtration before precipitation of the desired product to further insure purity. Furthermore, when R is isopropyl Step 4 is no longer necessary. Otherwise, however, the number of steps and their sequence is extremely critical to successful operation of this invention.

Thus applicant's discovery of the unique nature of isopropyl esters, and their facile transesterification has resulted in the novel combination of process steps detailed above which allows greater yield and product pu-

rity than known heretofore in the preparation of the subject phenoxybenzoic acid esters.

The following detailed examples are intended as illustrations rather than limitations on the scope of this invention so as to provide a better understanding of the nature, objects and advantages of the invention.

EXAMPLE 1

Preparation of Isopropyl 3-Chlorobenzoate

Isopropyl alcohol (66.0g, 1.1 mole) was added slowly to stirred 3-chlorobenzoyl chloride (175.0g, 1.0 mole) as the temperature was raised from 25° to about 85° C. A slight exotherm was controlled by adjustment of the addition rate. Most of the hydrochloric acid produced in the reaction was evolved during a 2-3 hr. reflux period. Excess isopropyl alcohol and residual hydrochloric acid were removed by application of an aspirator vacuum. The resulting pale yellow liquid product weighed 196.5g (99% yield-99% purity).

EXAMPLE 2

Preparation of Isopropyl 5-Chloro-2-Nitrobenzoate

Isopropyl 3-Chlorobenzoate (196.5g, 0.99 mole) was added dropwise to a −20° C stirred solution of 95% nitric acid (664g, 10.0 moles) over a 30 minute period. The temperature was maintained at −20° C for 3 hours and then raised to +20° C. About 80% of the nitric acid was removed by vacuum distillation giving a concentration of ca. 95% which can be directly reused in subsequent reactions. The residue was diluted with ice water (400 ml). The pale yellow solid precipitate was filtered and washed twice with water (100 ml). Purification was achieved by a sustained vacuum filtration is a sintered glass funnel for a period of about 24–30 hours to remove most of the oil. The dried solid amounted to 192.7g (80% yield -96% purity) and melted at 56°–58° C.

EXAMPLE 3

Preparation of Isopropyl 5-(2',4'-Dichlorophenoxy)-2-Nitrobenzoate

Solid potassium carbonate (60.5g, 0.44 mole) was added to a stirred solution of 2,4 dichlorophenol (135.5g, 0.83 moles) in dimethylformamide (DMF) (320 ml). The temperature was raised to reflux for 1 hour evolving carbon dioxide. Approximately one-half of the DMF solvent was removed by distillation along with the water formed in the reaction. Isopropyl 5-chloro-2-nitrobenzoate (192.5g, 0.79 moles) was added in one portion to the 150°–160° C stirred solution of the phenol salt. The reaction temperature dropped to ca. 120° C, but was raised back to 150° C. The reaction mixture was stirred for an additional 1 ½ hours during which time a large amount of potassium chloride precipitated. An aspirator vacuum was applied and 90% of the remaining DMF was removed by distillation. Upon cooling, the residue was suspended in water (500 ml) with vigorous stirring to give a finely divided yellow precipitate. The solid was filtered, washed twice with water (200 ml) and dried to give 266g (91% yield-93% purity) with a melting point of 54°–58°C.

EXAMPLE 4

Preparation of Methyl 5-(2',4'-Dichlorophenoxy)-2-Nitrobenzoate

A stirred solution of isopropyl 5-(2',4'-dichlorophenoxy)-2-nitrobenzoate (266g, 0.72 mole) and a catalytic amount of sodium methoxide (2.7g, 0.05 mole) in absolute methanol (560g, 18.0 moles) was refluxed for about 4 hours. Upon cooling the solution to 5° C a yellow solid precipitated. After stirring for 1 hour the solid was filtered and washed with low boiling petroleum either (200 ml). The dried solid amounted to 204g (83% yield-99% purity) and melted at 85°–87° C.

EXAMPLE 5

Preparation of Ethyl 5-(2',4'-dichlorophenoxy)-2-Nitrobenzoate

A procedure analogous to that of Example 4 was used through the intermediate steps (1–3*b*). Transesterification to the ethyl ester was accomplished with ethanol as reactant-solvent. The dried solid was obtained in 78% yield and 98% purity and melted at 83°–85° C.

EXAMPLE 6

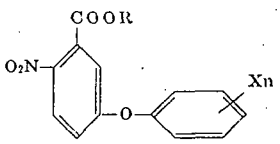
Preparation of Methyl 5-(2',4',6'-trichlorophenoxy)-2-Nitrobenzoate

A procedure analogous to Example 4 was used throughout. The analogous Example 3 intermediate, isopropyl 5-(2',4',6'-trichlorophenoxy)-2-nitrobenzoate, starting from 2,4,6-trichlorophenol was obtained in 65% yield and 95% purity after crystallization from petroleum ether and melted at 71°–74° C. Transesterification was with methanol in a procedure similar to that of Example 4. The solid product was obtained in 84% yield and 99% purity, melting at 101°–103° C.

Other disclosed compounds not exemplified in the examples are prepared in similar manner and are intended within the scope of this invention.

What is claimed is:

1. In a process for synthesizing phenoxybenzoic acid esters having the following general structure:



wherein R is alkyl (C_1–C_5), X is selected from the group consisting of hydrogen, iodine, fluorine, chlorine, bromine and combinations thereof, and *n* is an integer of from 2 to 5, the improvement which comprises (1) esterifying a halobenzoyl halide with isopropyl alcohol at a temperature from about 25° C to about 85° C to produce a halobenzoate ester, (2) nitrating the halobenzoate ester at a temperature of from about −50° to about +45° C with nitric acid in a mole ratio of nitric acid to ester substrate of between about 5:1 to about 20:1; (3) reacting the resulting isopropyl nitrobenzoate thereby produced with an alkali metal phenolate at a temperature of from about 70° to about 200° C to obtain an isopropyl halophenoxynitrobenzoate ester; (4) and, when the desired halophenoxynitrobenzoic acid ester is other than the isopropyl ester, transesterifying said isopropyl ester with a molar excess of a lower (C_1–C_5) alkyl alcohol other than isopropanol in the presence of a catalyst selected from the group consisting of alkali metals and alkali metal alkoxides to obtain the desired phenoxybenzoic acid ester.

2. The process of claim 1 where the halobenzoyl halide is selected from the group consisting of chloro-, and fluorobenzoyl chloride or fluoride and combinations thereof.

3. The process of claim 2 where the halide is m-chlorobenzoyl chloride.

4. The process of claim 1 where the alcohol contains from one to five carbon atoms.

5. The process of claim 4 where the alcohol is methanol.

6. The process of claim 1 where the desired halophenoxynitrobenzoic acid ester is methyl 5-(2',4'-dichlorophenoxy)-2-nitrobenzoate.

7. The method of claim 1 where the alcohol and catalyst are added directly to the reaction residue of Step 3 without recovering the isopropyl halophenoxynitrobenzoate ester.

8. The method of claim 1 where the halobenzoate ester in nitrated at about −20° to +45° C.

9. A method of making methyl 5-(2',4'-dichlorophenoxy)-2-nitrobenzoate comprising (1) esterifying m-chlorobenzoyl chloride with isopropyl alcohol at a temperature of from about 25° to about 85° C; (2) nitrating the resulting chlorobenzoate ester at a temperature from about −20° to about +20° C in nitric acid in a mole ratio of nitric acid to ester substrate of about 5:1 to 10:1; (3) reacting 2,4-dichlorophenolate with the nitrated halobenzoate ester from step (2) to produce isopropyl 5-(2',4'-dichlorophenoxy)-2-nitrobenzoate; (4) and transesterifying the isopropyl ester from step (3) with a molar excess of methyl alcohol in the presence of a catalyst selected from the group consisting of alkali metals and alkali metal alkoxides to obtain methyl 5-(2',4'-dichlorophenoxy)-2-nitrobenzoate.

* * * * *

LSC → I. MILES

United States Patent Office

3,816,526

Patented June 11, 1974

3,816,526

METHOD FOR PRODUCING HALOBENZOYL HALIDES

Anthony T. Jurewicz, Kendall Park, N.J., assignor to Mobil Oil Corporation

No Drawing. Filed Oct. 3, 1972, Ser. No. 294,750

Int. Cl. C07c 63/10

U.S. Cl. 260—544 M 9 Claims

ABSTRACT OF THE DISCLOSURE

Halogenation of benzoyl halide in the presence of a ferric halide catalyst and a halogenated hydrocarbon solvent provide higher yields of the meta isomer of monohalogenated benzoyl halide.

CROSS REFERENCE TO RELATED APPLICATIONS

None.

BACKGROUND OF THE INVENTION

Field of the invention

This invention is concerned with a process of making meta-halobenzoyl halides in improved yield which are highly useful starting materials in the production of certain herbicides, i.e., halophenoxynitrobenzoates.

For example, the process of this invention can be utilized in making the halophenoxy benzoic acid herbicides disclosed by U.S. Pat. 3,652,645.

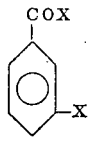
Description of the prior art

E. Hope and G. C. Reily reported in the Journal of the Chemical Society, vol. 121, pp. 2510, 1922 the catalytic halogenation of benzoyl halides. However, no solvents were used. Other methods that have been described in the prior art for the preparation of such halides generally start with materials that already contain nuclear halide. For example, the conversion of chlorobenzaldehyde to chlorobenzoyl chloride with carbon tetrachloride. In these processes the monohalogenated form produced contained meta-, ortho- and para-fractions.

Applicant, however, has discovered a means whereby the desired meta isomer of the mono-halogenated benzoyl halide is produced in higher yield than previously known to the art.

SUMMARY OF THE INVENTION

This invention provides, in a method for producing halobenzoyl halides having the following general structure:



where X is chlorine, bromine, fluorine, iodine or combinations of said halides, the improvement wherein a benzoyl halide is halogenated in the presence of a ferric halide catalyst and a halogenated hydrocarbon solvent whereby greater yields of the desired meta isomer are obtained.

DESCRIPTION OF SPECIFIC EMBODIMENTS

This invention, therefore, provides a process for synthesizing halobenzoyl halides wherein the desired meta isomer of the mono-halogenated fraction of said halide is preferentially produced in high yield. Accordingly applicant's process produces yields of the desired meta isomer substantially higher than, to the best of his knowledge, is known to the prior art.

The invention is specifically directed toward the halogenation of a benzoyl halide in the presence of both an anhydrous ferric halide and a halogenated hydrocarbon solvent. The combination of the ferric halide catalyst and the halogenated hydrocarbon solvent results in a greater selectivity to the meta isomer of the monohalogenated benzoyl compound during the halogenation.

Non-limiting examples of compounds conveniently prepared according to this invention are: chlorobenzoyl chloride; chlorobenzoyl fluoride; fluorobenzoyl fluoride; bromobenzoyl bromide; iodobenzoyl iodide; chlorobenzoyl bromide; iodobenzoyl bromide and the like. Preferred are meta-chlorobenzoyl chloride and meta-chlorobenzoyl fluoride.

Non-limiting examples of halogenated hydrocarbon solvents used are the normally liquid materials such as carbon tetrachloride, carbon tetrafluoride, carbon tetrabromide and such di-, tri- or tetrahaloalkanes (C_1–C_4) as dichloromethane, dibromomethane, dichloroethane, tribromomethane, tetrachloroethane and the like. Chlorinated hydrocarbon solvents especially carbon tetrahalides, e.g., carbon tetrachloride, are preferred solvents.

The concentration of the benzoyl halide in the solvent can range from 1 to 99 percent by weight of the total composition, however, the meta content of the monohalogenated benzoyl halide increases with increasing dilution of the benzoyl compound.

The catalyst system used is a ferric halide or a combination of a halogen and a ferric halide. Non-limiting examples are, ferric chloride, ferric bromide, anhydrous ferric chloride, anhydrous ferric bromine, anhydrous ferric iodide, or a combination of iodine and anhydrous ferric chloride, etc. Anhydrous ferric halides are preferred to prevent possible conversion of the benzoyl halide to its acid form by the formation of water during the reaction. Especially advantageous is the combination of a halide and the anhydrous ferric halide. Use of this combination can result in a two to three fold reaction rate increase and a further improved meta isomer distribution in the monohalogenated fraction. The concentration of the catalyst can vary from about 0.1 to about 5% weight based on the amount of benzoyl halide present with the preferred range being from about 0.2 to 3 weight percent. However, when the catalytic combination of, for example, anhydrous ferric chloride and iodine are used, the preferred weight ratio is 10 to 50 within this concentration range.

The reaction temperature can vary within wide limits from about 0° C. to the boiling point of the solvent used under the reaction conditions employed. The preferred temperature, for example, when carbon tetrachloride is the solvent depends interalia on the catalyst system used. When anhydrous ferric chloride is used, the preferred temperature range is from about 30° to about 60° C. and when the anhydrous ferric chloride-iodine combination is used the preferred range is from about 5° to 40° C.

The rate of halogen addition to the reaction zone can also vary within wide limits depending upon the particular solvent used and solubility of the halogen in that particular solvent.

The process embodied herein can be carried out at atmospheric pressure, but any convenient or desired pressure may be used.

A review of prior art known to applicant reveals that the best overall yield of the monohalobenzoyl halide is approximately 76% with the isomer distribution of the monohalogenated fraction yielding about 83% of the meta fraction. Utilizing applicant's invention, over 80% of the benzoylhalide can be converted to the monohalogenated form with up to 95% of said monohalogenated fraction being meta.

The following detailed examples are intended as illustrations rather than limitations on the scope of this invention so as to provide a better understanding of the nature, objects and advantages of the invention.

EXAMPLE 1

To a three neck flask 100 ml. of carbon tetrachloride, 1.1 gms. of anhydrous ferric chloride ad 70 gms. of benzoyl chloride were charged. Chlorine was bubbled through the solution of 100 ml./min. After 330 minutes at 30° C., 85% of the benzoyl chloride was chlorinated, 80% was monochlorinated and 5% dichlorinated. The monochlorinated material was 6.5% ortho, 91.5% meta and 2% para. The chlorinated materials, as also in the following examples, were identified by standard gas chromatography techniques.

EXAMPLE 2

Under conditions identical to Example 1, for comparison, a run without a solvent was used. After 360 minutes, 87% of the benzoyl chloride was converted to 78% monochlorobenzoyl chloride and 9% dichlorobenzoyl chloride. The isomer distribution of the monochlorinated material was 15% ortho, 83% meta, and 2% para. It is readily apparent from the comparison of Examples 1 and 2 that almost a 10% improvement is obtained in Example 1 where a halogenated hydrocarbon solvent is used.

EXAMPLE 3

To a three neck flask 100 ml. of carbon tetrachloride, 1.1 gms. of anhydrous ferric chloride, 0.1 gm. iodine and 70 gms. of benzoyl chloride were added. Chlorine was bubbled through the solution at 100 ml./min. After 150 minutes at 10° C., 70.5% monochlorinated and 9% dichlorinated benzoyl chloride were obtained. The isomer ratio of the monochlorinated fraction was 3% ortho, 95% meta and 2% para.

EXAMPLE 4

To a three neck flask 100 ml. of dichloroethane, 1.1 g. of anhydrous ferric chloride and 7 g. of benzoyl chloride were charged. Chlorine was bubbled through the solution at 100 ml./min. After 370 minutes at 35° C., 63% monochlorinated and 2% dichlorinated benzoyl chloride were obtained. The isomer ratio of the monochlorinated form was 12% ortho, 86% meta and 2% para.

EXAMPLE 5

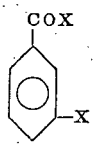
For comparison, a run identical to that of Example 1 was made except that 100 ml. of a non-halogenated solvent (octane) was used. After 360 minutes 26% monochlorinated benzoyl chloride was obtained. The isomer ratio was 18% ortho, 79% meta and 3% para. This example clearly demonstrates the advantages of using a halogenated hydrocarbon solvent to obtain the desired meta isomer.

In general the examples clearly demonstrate the advantages of using applicant's ferric halide-halogenated solvent system to obtain greater yields of the meta-isomer of a monohalogenated benzoyl halide.

Although the present invention has been particularly described with respect to preferred embodiments, all the disclosed embodiments and modifications apparent to one of ordinary skill in the art are considered to be within the scope of this invention.

What is claimed is:

1. In a method for producing a halobenzoyl halide having the following general structure:



wherein X is chlorine, bromine, fluorine, iodine or combinations of said halides, the improvement wherein benzoyl halide is halogenated in the presence of a halogen, an anhydrous ferric halide catalyst and a di-, tri- or tetrahaloalkane (C_1–C_4) halogenated hydrocarbon solvent.

2. The method of claim **1** where X is chlorine and the halobenzoyl halide is meta-chlorobenzoyl chloride.

3. The method of claim **1** where the catalyst is selected from anhydrous ferric chloride, or anhydrous ferric chloride in combination with iodine.

4. The method of claim **3** where the catalyst system is iodine and anhydrous ferric chloride.

5. The method of claim **4** where the halogenation is carried out at a temperature of from about 5° to about 40° C.

6. The method of claim **1** where the solvent is a chlorinated solvent.

7. The method of claim **6** where the solvent is carbon tetrachloride.

8. The method of claim **1** wherein the concentration of the catalyst is from about 0.1 to about 5% weight based on the amount of benzoyl halide present.

9. In a method for producing meta-chlorobenozyl chloride, the improvement wherein benzoyl chloride is chlorinated in the presence of chlorine, an anhydrous ferric chloride-iodine catalyst with carbon tetrachloride as the solvent.

References Cited

UNITED STATES PATENTS

2,890,243	6/1959	Brown et al.	260—544 M
3,014,965	12/1961	Newcomer et al.	260—544 F
3,096,367	7/1963	Newcomer et al.	260—544 M

LORRAINE A. WEINBERGER, Primary Examiner

R. D. KELLY, Assistant Examiner

U.S. Cl. X.R.

260—544 F

V-M102 GAGE TABLE
SERVICE-PRODUCT SURGE TANK
DIMENSIONS-10' DIA. X 22'L
100% = 21'

11.35 #/GAL

%	GALLONS	%	GALLONS	%	GALLONS	%	GALLONS
-1/2	62	27	3331	53-1/2	6600	80	9870
1	123	27-1/2	3393	54	6662	80-1/2	9931
1-1/2	185	28	3454	54-1/2	6724	81	9993
2	247	28-1/2	3516	55	6785	81-1/2	10,055
2-1/2	308	29	3578	55-1/2	6847	82	10,116
3	372	29-1/2	3639	56	6909	82-1/2	10,178
3-1/2	432	30	3701	56-1/2	6970	83	10,240
4	493	30-1/2	3763	57	7032	83-1/2	10,301
4-1/2	555	31	3824	57-1/2	7094	84	10,363
5	617	31-1/2	3886	58	7155	84-1/2	10,425
5-1/2	679	32	3948	58-1/2	7217	85	10,486
6	740	32-1/2	4010	59	7279	85-1/2	10,548
6-1/2	802	33	4071	59-1/2	7341	86	10,610
7	864	33-1/2	4133	60	7402	86-1/2	10,672
7-1/2	925	34	4195	60-1/2	7464	87	10,733
8	987	34-1/2	4256	61	7526	87-1/2	10,795
8-1/2	1049	35	4318	61-1/2	7587	88	10,857
9	1110	35-1/2	4380	62	7649	88-1/2	10,918
9-1/2	1172	36	4441	62-1/2	7711	89	10,980
10	1234	36-1/2	4503	63	7772	89-1/2	11,042
10-1/2	1295	37	4565	63-1/2	7834	90	11,103
11	1357	37-1/2	4626	64	7896	90-1/2	11,165
11-1/2	1419	38	4688	64-1/2	7957	91	11,227
12	1480	38-1/2	4750	65	8019	91-1/2	11,288
12-1/2	1542	39	4811	65-1/2	8081	92	11,350
13	1604	39-1/2	4873	66	8142	92-1/2	11,412
13-1/2	1665	40	4935	66-1/2	8204	93	11,473
14	1727	40-1/2	4996	67	8266	93-1/2	11,535
14-1/2	1789	41	5058	67-1/2	8327	94	11,597
15	1851	41-1/2	5120	68	8389	94-1/2	11,658
15-1/2	1912	42	5182	68-1/2	8451	95	11,720
16	1974	42-1/2	5243	69	8513	95-1/2	11,782
16-1/2	2036	43	5305	69-1/2	8574	96	11,844
17	2097	43-1/2	5367	70	8636	96-1/2	11,905
17-1/2	2159	44	5428	70-1/2	8698	97	11,967
18	2221	44-1/2	5490	71	8759	97-1/2	12,029
18-1/2	2282	45	5552	71-1/2	8821	98	12,090
19	2344	45-1/2	5613	72	8883	98-1/2	12,152
19-1/2	2406	46	5675	72-1/2	8944	99	12,214
20	2467	46-1/2	5737	73	9006	99-1/2	12,275
20-1/2	2529	47	5798	73-1/2	9068	100	12,337
21	2591	47-1/2	5860	74	9129		
21-1/2	2652	48	5922	74-1/2	9191		
22	2714	48-1/2	5983	75	9253		
22-1/2	2776	49	6045	75-1/2	9314		
23	2838	49-1/2	6107	76	9376		
23-1/2	2899	50	6169	76-1/2	9438		
24	2961	50-1/2	6230	77	9500		
24-1/2	3023	51	6292	77-1/2	9561		
25	3084	51-1/2	6254	78	9623		
25-1/2	3146	52	6415	78-1/2	9685		
26	3208	52-1/2	6477	79	9746		
26-1/2	3269	53	6539	79-1/2	9808		

Phone: (501) 572-3701 P. O. Box 2648

Eagle River Chemical Corp.

Highway 242
WEST HELENA, ARKANSAS 72390
January 5, 1975

C.E. Hunt
Mobil Chemical Co.
Edison Laboratories
P.O. Box 240
Edison, New Jersey 08817

Dear Sir:

At the request of John Ellington, we are forwarding to you under separate cover, 5 gallons of NBE assay 83% wt. The NBE sample was taken from material shipped by tank truck trailor #119 to Mt. Pleasant on January 3, 1976.

Please let us know if we can be of further help.

Thank you,

George F. Mather

CC: J.W. Shackelford
John Miles
Joe Porter/Larry Conaway
John Ellington

VELSICOL CHEMICAL CORPORATION

4902 CENTRAL AVE., · CHATTANOOGA, TENN. 37410 · AREA CODE 615 · 821-6521

November 11, 1975

Mobil Chemical
Mt Pleasant
Tennessee

TC Metachloro Methylbenzoate
MOBX 11391 shipped 11/6/75

VCC Order 84264
Customer Order M 9000

ANALYSIS

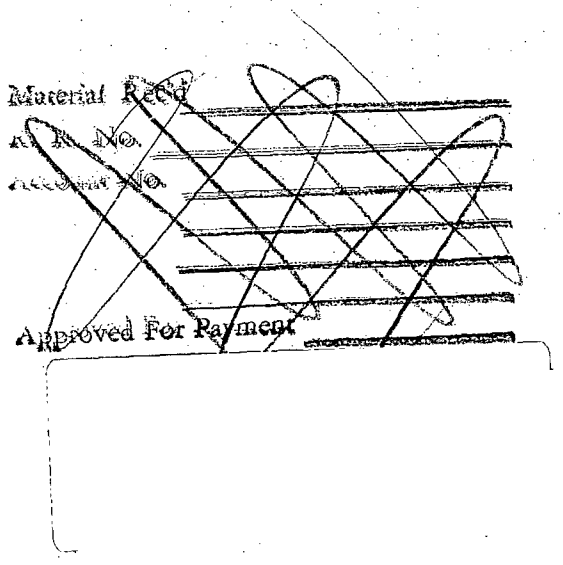
1.7% Methyl Benzoate
1.3% para-Chloromethyl Benzoate
93.7% meta-Chloromethyl Benzoate
3.3% ortho-Chloromethyl Benzoate

VELSICOL CHEMICAL CORPORATION

H. F. Price
Chief Chemist

sn

cc: Eagle River Chemical
West Helena, Arkansas



Phone: (501) 572-3701 P. O. Box 2648

Eagle River Chemical Corp.

Highway 242
WEST HELENA, ARKANSAS 72390
OCTOBER 24, 1975

TO: DON FAGGERT
SUBJECT: MOBIL NITRATION PROJECT STATUS REPORT

	%COMPLETE	COMPLETE	REMARKS
I. CONSTRUCTION			
PIPING	99+		
ELECTRICAL	75		
INSTRUMENTATION	40		
SAFETY EQUIPMENT	95		
UTILITY STATIONS	100		
ROOF	95		

II. MANPOWER
REQUIREMENTS ARE DECREASING DAILY.

III. COMMENTARY
ALL EFFORTS ARE NOW BEING CONCENTRATED IN THE AREA OF INSTRUMENTATION AND ELECTRICAL.

DELIVERY PROBLEMS HAVE ARISEN BUT DO NOT SEEM TO BE CRITICAL AS YET.

PUNCH LISTING OF THE PLANT IS BEING CONDUCTED DAILY.

Jim E. Fowler
JIM E. FOWLER
PROJECT MANAGER

CC: J.W. SHACKELFORD
RAY GUIDI
BOB FABIAN
GEORGE MATHER

MOBIL NITRATION PROJECT COST ESTIMATE

PREPARED SEPTEMBER 24, 1975

I.	MATERIAL & EQUIPMENT COSTS (TO DATE)		345,615
	FREIGHT & TOOLS		5,414
			351,029
II.	LABOR (TO DATE)		33,055
	EAGLE RIVER	3,055	
	CCI	30,000	
		33,055	
III.	ADDITIONAL ESTIMATED COSTS		40,000
	PIPE	10,000	
	ELECTRICAL	10,000	
	PAINT (INCLUDES LABOR)	8,000	
	INSULATION	12,000	
		40,000	
IV.	ESTIMATED LABOR TO COMPLETION		150,000
	BASIS: 50 men X 10 hr. X 6 day X $11 X 4wk=132M (SAY 150,000)		
			$574,084
	TOTAL COST	SAY	$575,000

V. VARIANCE FROM ORIGINAL $450,000 ESTIMATE

MAJOR ITEMS:

LABORATORY EQUIPMENT	5,000
SPUR INSTALLATION @ RR	21,000
ENGINEERING	30,000
ELECTRICAL RELOCATION	2,000
	$58,000

cc: RAY GUIDI
J.W. SHACKELFORD
BOB FABIAN
GEORGE MATHER
JOHN MILES
JOHN HOLCOMB

Jim E. Fowler

JIM E. FOWLER
PROJECT ENGINEER

September 19, 1975

To: Don Faggert
Subject: Mobil Nitration Project Status Report

	%Complete	Complete	Remarks
I. ENGINEERING			
Piping	100	9/19/75	
Instrumentation	95		
Electrical	100	9/15/75	
II. CONSTRUCTION			
Tank Form Dike	100	9/18/75	
Structural Steel Eraction			
Building Columns	80		
Intermediate Bracing	40		
Trusses	To be delivered 9/29/75		
Platform Structure	To be delivered 9/29/75		
Unloading Platforms	Delivered	9/19/75	
Equipment Installation			
V-M101 Solvent Stripper		9/18/75	Set
Support		9/18/75	Set
T-M104 Extraction Tank		9/18/75	Set
Support		9/18/75	Set
T-M109 Structural Steel		9/18/75	Set
E-M104 Still Heater		9/17/75	Set
Pipe Rack Structures	10		
Electrical			UNCHANGED
III. OFFSITES	Making utility taps 9/20/75		
IV. EQUIPMENT	See Attached		

V. COMMENTARY

Piping engineering is complete at this time and fabrication is underway.

Instrumentation purchases are complete and no delivery problem is anticipated.

Much of the structural bottleneck has been eliminated this week and critical work should be unhampered.

Anticipate beginning night time pipe fabrication next week.

Man power availability has yet to present itself a problem.

Jim E. Fowler

JIM E. FOWLER
PROJECT MANAGER

CC: J.W. Shackelford
Ray Guidi
Bob Fabian
John Holcomb
George Mather

MOBIL EQUIPMENT STATUS

11 Aug 75

Exchanage	Nomenclature	Vendor	P.O. #	Ordered	Shipped	Received
E-M101	Nitration Reaction Cooler	La Chem Equip	2388	6 Aug 75	12 Aug 75 Younger Bros	15 Aug 75
E-M102	Still Condenser	La Chem Equip	2388	6 Aug 75	12 Aug 75	15 Aug 75
E-M103	Still Tail Condenser	La Chem Equip	2388	6 Aug 75	12 Aug 75	15 Aug 75
E-M104	Still Heater	La Chem Equip	2388	6 Aug 75	12 Aug 75	15 Aug 75
E-M105	Surface Condenser & J-M101 A & B	S & K	2425	7 Aug 75	17 Oct 75	
E-M106	TW Heater	Denver	~	~	~	On Site
TANKS						
T-M101	H_2SO_4 Day Tank	Holland		8 Aug 75	1 Sept 75	2 Sept 75
T-M102	HNO_3 Day Tank	Denver	~	~	~	On Site
T-M103	EDC Day Tank	Denver	~	~	~	On Site
T-M104	Extraction Tank	Denver	~	~	~	On Site
T-M105	Spent Acid Hold Tank	Holland		8 Aug 75	1 Sept 75	2 Sept 75
T-M106	$(NH_4)_2SO_4$ Waste Tank	Holland		8 Aug 75	1 Sept 75	2 Sept 75
T-M107	Aqua/NH_3 Blend Tank	Denver	~	~	~	On site
T-M108	HNO_3 Storage Tank	Holland	2328		1 Oct 75	
T-M109	Sump					@ Site
T-M110	H_2SO_4 Storage Tank	Holland			15 Sept 75	22 Sept 75
VESSE						
R-M101	Nitration Reactor	Fed Equip Co	2355	4 Aug 75	12 Aug 75	15 Aug 75
Y-M101	Solvent Stripper	La Chem Equip	2389	6 Aug 75	12 Aug 75	15 Aug 75
Y-M102	Product Surge Tank	Holland		8 Aug 75	15 Sept 75	19 Sept 75
PUMPS						
P-M101	98% H_2SO_4 Metering	Jack Tyler	2390	8 Aug 75	1 Sept 75	
P-M102	97% HNO_3 Metering	Jack Tyler	2390	8 Aug 75	1 Sep 75	
P-M103-115		Denver				On Site
ELECTR						
E-MCC-120	Motor Control Center	Refrig Elec Sup	2510	14 Aug 75	17 Sept 75	

Phone: (501) 572-3701

P. O. Box 2648

Eagle River Chemical Corp.

Highway 242

WEST HELENA, ARKANSAS 72390

Mr. John Ellington
P.O. Box 352
Mt. Pleasant, Tenn 38474

Dear John;

A weekly process control graph for the NBE unit will be sent to you indicating NBE daily production, Molar yields, purity % NBE (V-M102), % EDC (V-M102), % CBE (R-M101), % NBE in spent acid, % NBE in ammonium sulfate. The graphs enclosed are from start up to December 13. Monthly, we will send a complete graph, for that month, on the yearly form you supplied us.

As you are aware, our NBE purity analysis has been a problem for us and the graph attached reflects our analytical variation. Larry Conaway and Joe Porter assure me that resolution is forthcoming.

Please let us know if we can be of further assistance.

Yours truly,

George Mather

GFM/rvk
Enclosure (3)

CC: Don Faggert
Bob Fabian
Larry Conaway
John Miles
George Mather
J.W. Shackelford
Jim Fowler

Raw Materials - Mobil would supply all raw materials. The CBE would arrive via tank car and would be used as storage for the process. Mobil would also supply their own tank car fleet for shipment of the NBE finished product. Therefore, our finished tankage would be limited to a small surge tank. The raw material valuation per batch is in the neighborhood of $ 15,000 - $ 20,000/1,500 gal. batch. This will require a much more efficient and tighter operation than our present plant practices at Eagle River. The CBE specification is 95% CBE, 0.5% methanol, and 0.5% dichloro-CBE. The remainder consists of the para- and ortho-isomers. Steam will be required thawing and unloading of CBE with nitrogen pad pressured okayed for unloading. Mobil will supply the ethylene dichloride under their own purchasing arrangements, but we will need to check incoming for methanol content. Methanol build-up is a problem in the overall process and has to be monitored. The NBE will slowly hydrolize to methanol and will interfer with the reaction. Mobil recommended teflon gaskets on all EDC handling equipment. Mobil will supply 97% nitric acid and 98% sulfuric acid in bulk. We would use existing storage for these products. The EDC would be supplied in a small bulk shipment for startup and then would be augmented from drums. The other raw material would be aqua ammonia supplied in bulk with a couple of small gaseous cylinders for touch-up additions.

Operations - Mobil strongly recommends that the refrigeration system use calcium chloride brine rather than the organic refrigerants that we are planning to use in Product 10. Based on their recommendations and the high cost of a bad batch, I would recommend that we give this serious consideration. Agitation requirements should be sufficient for an average specific gravity of 1.6 and cooling capacity should be adequate for a heat requirement of 430 B.T.U. per pound of CBE. Good agitation is essential for safe operation as well as yield performance. If agitation stops during nitric addition, it is necessary to stop the reaction, phase separate and discharge to a holding tank. Dinitro formation is possible under these conditions. The reaction goes to completion fairly well when impurities are not present. Nitric is added at 1% excess over molar quantities. The finished product should not contain in excess of 1% CBE. 32^{0} F. temperature is necessary for good yield of the correct isomer. Lower temperatures increase the isomer distribution favorably. Mobil recommends tempered H_2O for heatup at the end of the reaction. Extraction is necessary to recover the NBE from the spent sulfuric acid. 10 - 12% NBE is normally left in the spent sulfuric which ranges from 80 - 83% sulfuric. The efficientcy of the extraction step depends on the EDC recycle quality. The recycled EDC runs approximately 8% NBE per Mobil's experience. After extraction, 1 - 3% NBE in the acid is considered good performance and 1 - 2% EDC is also expected performance. The spent acid is then shipped back to Mobil in tank cars. Aqua ammonia is used for the neutralization of the NBE solution prior to the stripping operation. The neutralization is a fairly simple operation with approximately 200 gallons of 35% ammonium sulfate solution. We will supply a tank and Mobil will take the product in bulk-either tank trucks or tank cars. We should have no effluent from this plant operation if we arrange to recycle the steam jet condensate back into the neutralization step as Mobil currently does. The aquaeous ammonium sulfate will contain 3 - 5% organic as EDC and NBE which are not readily biodegrable. However, this could be blended with purer materials and resold

as fertilizer solution. The separations are very important and Mobil suggests the use of a small side tank for collection and recycle of the interface.

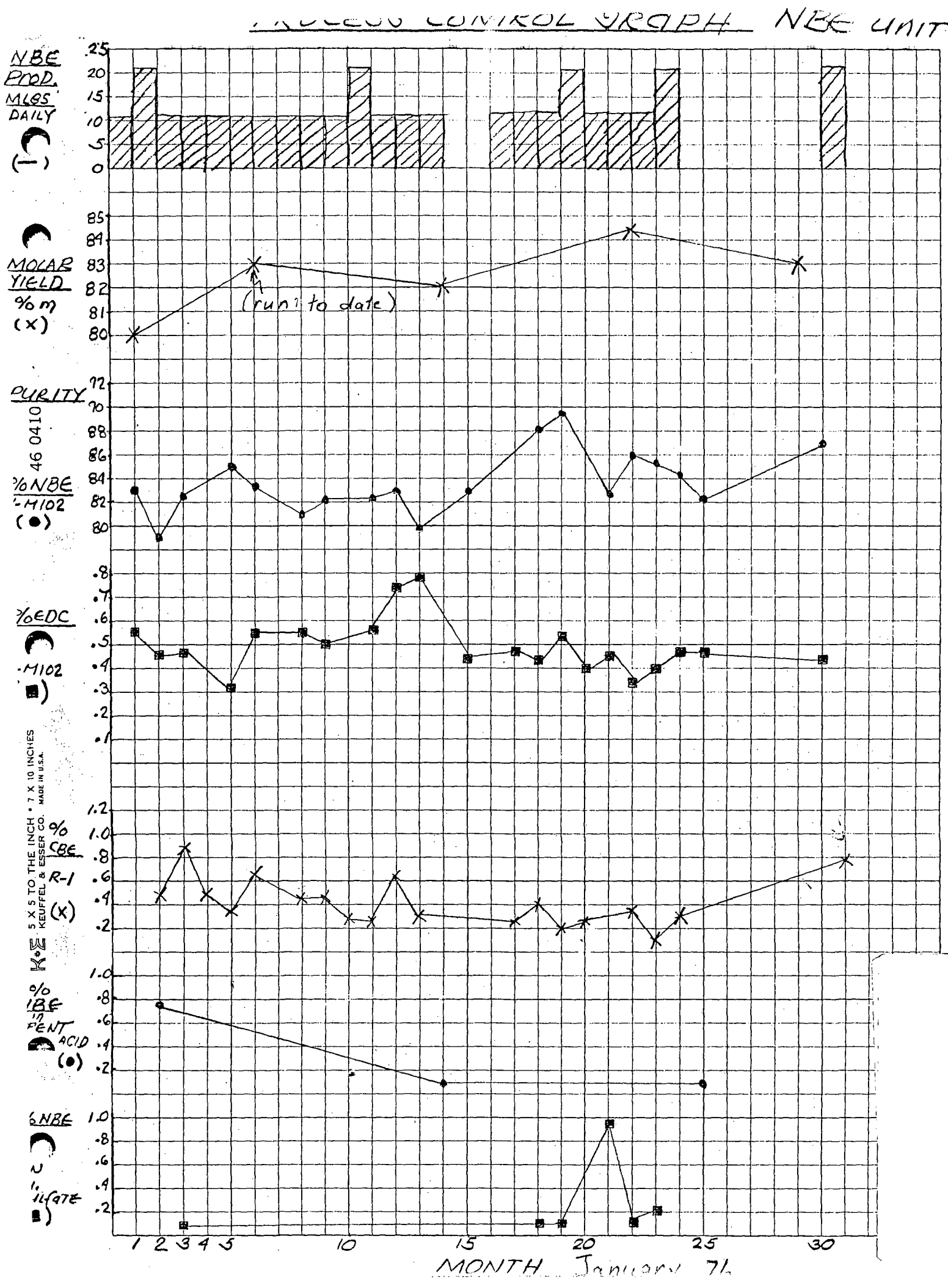
The vacuum source should be capable of 30 mm of mercury. The stripping operation should be capable of operating at 275° F. Mobil recommended two stage cooling with water for cooling to 150 mm of mercury and brine cooling for the end point. They report no product degradation to 300° F. and strip to 0.5% EDC which is their shipping specification. Their typical finished product assay is 85 - 86% NBE, 2 - 3% CBE, 0.5% EDC. The remainder is the other NBE isomers. The NBE has a freezing point of 118° F. And, therefore, requires good steam tracing on all handling after the stripping stage.

Process Controls - Phase separations are most critical in this process. Sloppy phase separations will inhibit yield as well as the reaction completion. Mobil recommends sight glasses and good methods for interface recognition and decreased flow rates to make the separations clean.

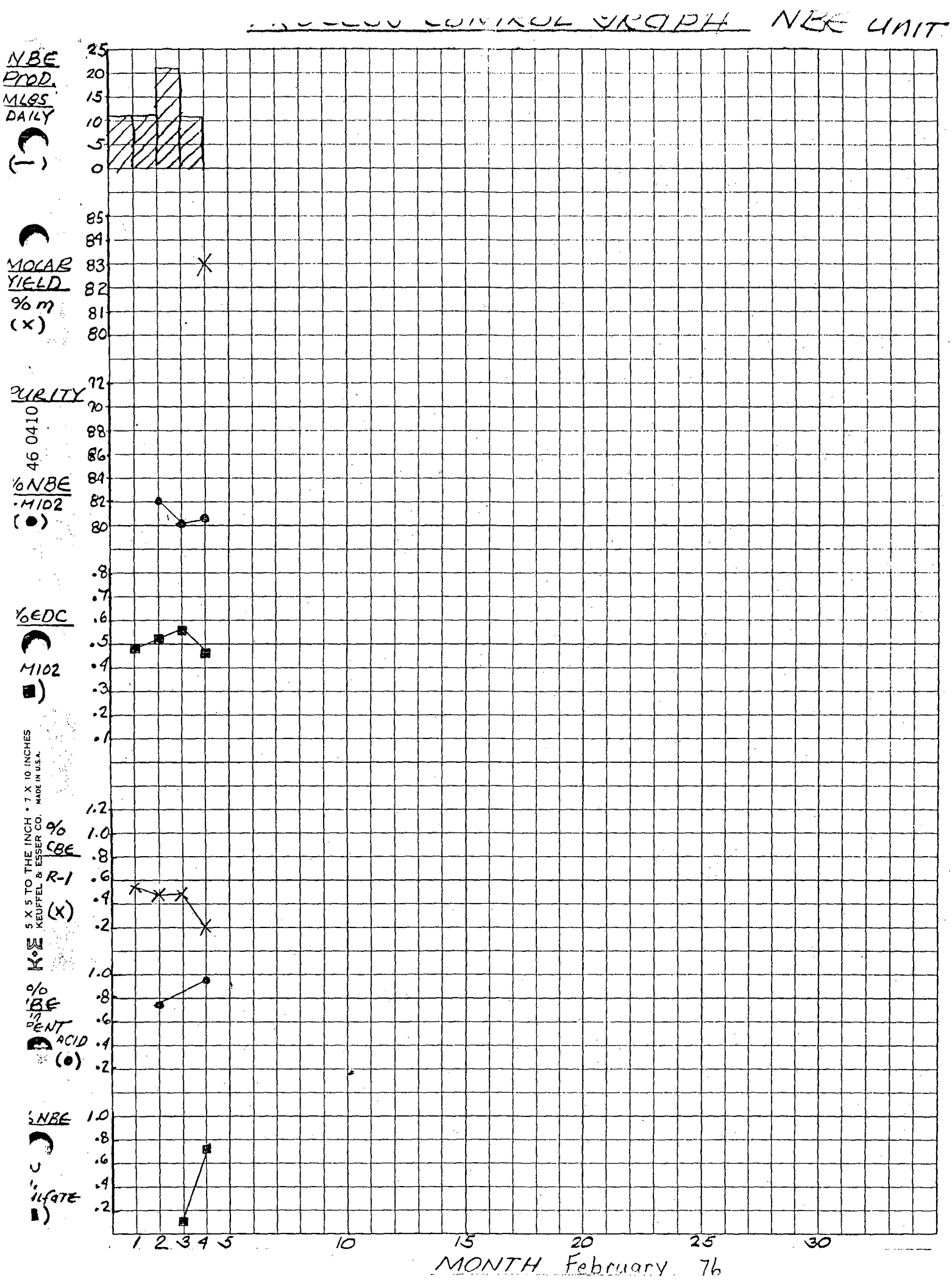
Mobil uses dual temperature indicator control on their reactors interlocked to the cut-off to stop acid flow at temperatures above 37°. They recommend electrical controls for fast response. They recommend positive displacement metering, such as a Viking pump to properly control acid feed rate. They indicate that the reaction tends to level out at about 1/3 nitric addition and can show a sudden upsurge. Good agitation is essential for yield as well as minimizing the runaway potential. Mobil recommends a turbon type agitator with approximately a 10 H.P. mixer which incorporates an ammeter indicator interlock to warn of an overload prior to a breaker stoppage of the agitator. In case of power outage for more than 5 seconds, the acid flow and the agitation is interlocked to stop and will not restart. To prevent operator mistakes, Mobil recommends the use of selector switches before pumps will operate.

It is recommended that the acid be added to the circulating line in front of the pump and cooler. Also, recommended that the aqua ammonia be added to the circulating line, and by pass the heating exchanger. They also recommend that a strainer be used to catch any ammonium sulfate from the stripping operation. They also recommend that the EDC tank be checked regularly for ammonium sulfate as well as H_2O buildup.

PROCESS CONTROL GRAPH NBE UNIT



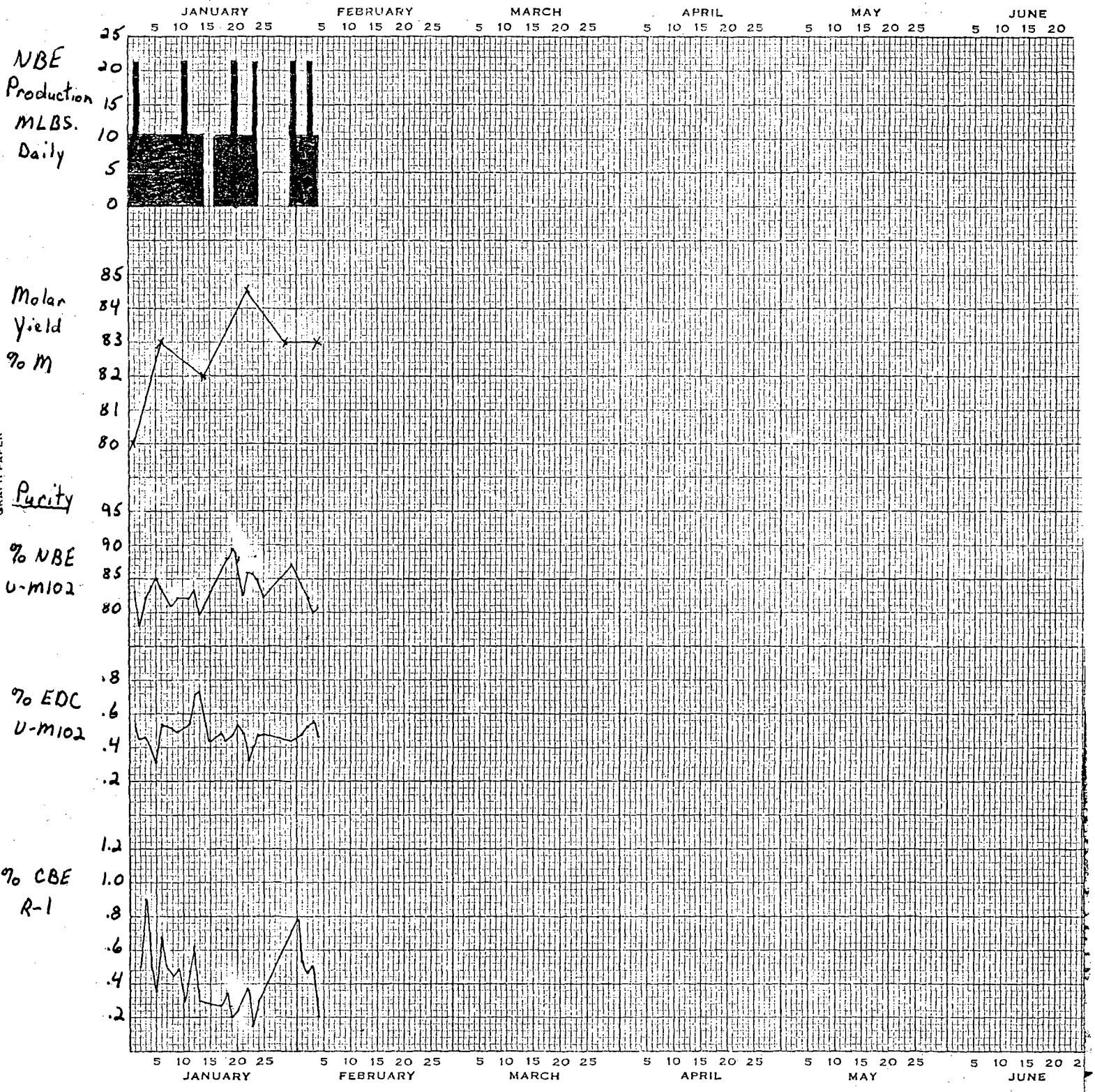
PROCESS CONTROL GRAPH NBE UNIT



Process Control Graph

NBE Unit

1976



MOBIL NBE YIELDS

PERIOD 1/1 - 2/1/76

MATERIAL	ACTUAL YIELD LB/LB NBE	TARGET YIELD PER MOBIL LB/LB NBE
NH_3	.038	.044
HNO_3	.339	.357
H_2SO_4	.874	.877
EDC	.104	.0996
CBE	.823	.82

Phone: (501) 572-3701

P. O. Box 2648

Eagle River Chemical Corp.

Highway 242

WEST HELENA, ARKANSAS 72390

June 4, 1975

Don Faggert
Manager, Corp. Chemical Formulation
Mobil Chemical Company
P.O. Box 26683
Richmond, VA 23261

Dear Don,

Enclosed are preliminary flow diagram and a revised equipment list for Mobil's comments. I will have a more complete diagram at the meeting scheduled for June 10, but I thought your people would like to have an earlier idea of the direction we are taking.

Sincerely,

John Miles
Project Manager

EQUIPMENT REQUIREMENT-MOBIL NITRATION-REVISED

I. VESSELS

1. 97% Nitric Acid Day Tank - 300 gal, 304SS
2. 98% Sulfuric Acid Day Tank - 600 gal. CS
3. EDC Day Tank - 2500 gal, CS
4. Reactor - 3000 gal. 316SS Atmospheric w/15HP Turbine Agitator
5. Extraction Tank - 2000 gal. 316SS w/Agitator
6. Spent Acid Hold Tank - 8000 gal. CS
7. $(NH_4)_2SO_4$ Waste Hold Tank - 5000 gal. CS
8. Product Still/EDC Stripper - 3000 gal. 316SS Full Vacuum Rated
9. Ejector Hot Well - 500 gal. CS
10. Product Surge Tank - 2000 gal. CS w/Steam Coil or Jacket

II. HEAT EXCHANGERS

1. Reactor Cooler - 600 ft^2 316SS Tube CS Shell
2. Still Heater - 400 ft^2 304SS 75 psig Shell Side
3. Still Condensers - A) 100 $ft.^2$ CW Condenser CS Full Vacuum Rated
   B) 100 $ft.^2$ Brine Tail Condenser CS Full Vacuum Rated

III. PUMPS

0) Waste Pumps (2-100GPM)
1) 97% HNO_3 Feed Pump-Metering Type Pump-0-2 GPM
2) 98% H_2SO_4 Feed Pump-Metering Type Pump-0-3 GPM
3) EDC Day Tank Pump-40 GPM @ 80'-2x1 CS
4) Reactor Circulation Pump-500 GPM @ 130'-4x3 316SS
5) Reactor Phase Seperation Pump-20 GPM @ 60'-1½x1 316SS
6) Extraction Tank Pump-40 GPM GPM @ 80'-2x1 316SS
7) Still Circulation Pump-250 GPM @ 120'-3x2 316SS
8) Product Surge Tank Pump-100 GPM @ 100'-3x1½

IV. MISCELLANEOUS

1) Vacuum System - 2-Stage-Non-condensing Steam Ejector Set
2) In-Line Static Mixer

V. TOTAL EQUIPMENT

(5) In any event, your obligations of confidence under this agreement shall terminate five years from the date of acceptance set forth herein below.

Very truly yours,

MOBIL CHEMICAL COMPANY
A Division of Mobil Oil Corporation

By: _____
J. P. Rogers
Vice President & General Manager

ACCEPTED AND AGREED

By: _____

Date: _____

MOBIL NITRATION PROJECT COST ESTIMATE

PREPARED SEPTEMBER 24, 1975

I.	MATERIAL & EQUIPMENT COSTS (TO DATE)		345,615
	FREIGHT & TOOLS		5,414
			351,029
II.	LABOR (TO DATE)		33,055
	EAGLE RIVER	3,055	
	CCI	30,000	
		33,055	
III.	ADDITIONAL ESTIMATED COSTS		40,000
	PIPE	10,000	
	ELECTRICAL	10,000	
	PAINT (INCLUDES LABOR)	8,000	
	INSULATION	12,000	
		40,000	
IV.	ESTIMATED LABOR TO COMPLETION		150,000
	BASIS: 50 men X 10 hr. X 6 day X $11 X 4wk=132M (SAY 150,000)		
			$574,084
	TOTAL COST	SAY	$575,000

V. VARIANCE FROM ORIGINAL $450,000 ESTIMATE

MAJOR ITEMS:

LABORATORY EQUIPMENT	5,000
SPUR INSTALLATION @ RR	21,000
ENGINEERING	30,000
ELECTRICAL RELOCATION	2,000
	$58,000

cc: RAY GUIDI
J.W. SHACKELFORD
BOB FABIAN
GEORGE MATHER
JOHN MILES
JOHN HOLCOMB

Jim E. Fowler

JIM E. FOWLER
PROJECT ENGINEER

Phone: (501) 572-3701

P. O. Box 2648

Eagle River Chemical Corp.

Highway 242

WEST HELENA, ARKANSAS 72390

December 15, 1975

To: J.W. Shackelford

From: Jim E. Fowler

Subject: Mobil Nitration Costs - 195M

I.	Total of entries in account 195M	$604,147.88
	Value of surplus inventoryy	44,187.13
	Mobil Capital Cost	$560,060.75
II.	Estimate dated 24 Sept 75	$575,000.00
	Cost (actual)	560,060.75
	Variance	$ 14,939.25

III. Composite Itemization

Labor: Manhours 18,293

CCI	$113,989.50
DUNLAP ELECTRIC	9,665.75
*NORHT BROTHERS (INSULATION)	13,262.52
PLANT FORCES @ EAGLE RIVER	16,540.28
*DELK MASONRY	2,900.00
Total Labor	$156,358.05 = $8,547/Mhr

* Includes Material

Engineering

Shreeve Engineering Ltd	$ 45,587.00
*Instrumentation (Pocsik)	2,932.00
Total Engineering	$ 48,519.00

* Includes expenses

Material and Equipment $355,183.70

Phone: (501) 572-3701

P. O. Box 2648

Eagle River Chemical Corp.

Highway 242

WEST HELENA, ARKANSAS 72390

December 15, 1975

IV. Commentary

A considerable number of overtime hours were utilized to complete the project within the Mobil Chemical time requirements, resulting in higher labor costs per man hour. The construction was begun on August 8, 1975, and the first batch of NBE was produced on November 20, 1975. This represents a total of 84 days of construction time.

An insignificant number of difficulties have occured since production has begun, but there is a list of items within the Mobil unit that require alternation or addition to facilitate more efficient production. It is my estimate that those revisions can be made at a cost that should not exceed $20,000.00. Eagle River personnel should be able to perform this work in approximately one week. A good target period to do this work is immediately after delivery of 500,000 pounds of NBE required by January 31, 1976. Accomplishment of this extra work and inclusion of extra control equipment will make the unit easier to operate and should provide better optimization of utilities; and promote additional unit safety.

Jim E. Fowler

Jim E. Fowler
Project Manager

CC: Ray Guidi
Bob Fabian
George Mather
John Miles
John Holcomb
Larry Conaway

VERTAC
INCORPORATED
Building 4—Suite 412 • Shreve City Office Park • Shreveport, Louisiana 71105 • Phone 318-869-3434

July 28, 1975

Mr. Richard J. Goertz
Vice President - General Manager
Mobil Chemical Company
P. O. Box 26683
Richmond, Virginia 23261

Dear Mr. Goertz:

Enclosed is a copy of your letter dated July 21, 1975 which I have executed on behalf of Vertac, Inc.

We are sincerely grateful to you and your people for your confidence in the Vertac group, as expressed by your placing with us your needs for NBE. Please be assured that our people will make every effort to erect the necessary facilities. Our Eagle River group has confirmed to me that they will meet your delivery requirements for product.

Sincerely,

R. A. Guidi

RAG-el

Encl.

cc: Bill Shackelford
George Mathen

Mobil Chemical Company

CROP CHEMICALS GROUP

P.O. BOX 26683
RICHMOND, VIRGINIA 23261
TELEPHONE (804) 798-4291

July 21, 1975

Mr. R. A. Guidi
Vice President, Manufacturing
and Engineering
Vertac, Incorporated
Building 4, Suite 412
Shreve City Office Park
Shreveport, Louisiana 71105

Dear Mr. Guidi:

You will find enclosed a copy of the agreement we have prepared for the manufacture of NBE.

We understand that in order for your Eagle River subsidiary to be ready to begin manufacturing in November, it will be necessary for work to begin promptly on certain changes in and additions to Eagle River's facilities. In consideration for your causing this work to begin promptly and to be carried forward expeditiously without waiting for execution of the enclosed contract, Mobil agrees that if Vertac in good faith attempts to negotiate and to execute before October 1, 1975 an agreement in substantially the form of the enclosed draft contract and if because of a refusal by Mobil to negotiate or execute such a contract no agreement concerning the manufacture of NBE is executed by October 1, 1975, then Mobil will reimburse Eagle River for the direct engineering, procurement, and construction costs and expenses incurred by it in making the physical changes required to manufacture NBE, which costs and expenses will not exceed $100,000.00 without Mobil's prior written consent.

Please confirm our agreement regarding these start-up costs by signing and returning the enclosed copy of this letter.

Very truly yours,

Richard J. Goertz
Vice President - General Manager

Vertac, Incorporated

By: _____

VERTAC CONSOLIDATED
A MEMBER OF THE VERTAC GROUP

SUITE 2414 • 5100 POPLAR • MEMPHIS, TN 38137 • 901-767-6851 • TELEX 53927

October 26, 1976

Mr. Don Faggert
Mobil Chemical Company
P. O. Box 26683
Richmond, Virginia 23261

Re: Agreement of September 1, 1975

Dear Don:

As discussed in our meeting in Memphis, October 14, 1976, we propose that the referenced agreement between Mobil Chemical Company and Vertac, Incorporated be amended effective January 1, 1977, to provide for an extension of the agreement from March 31, 1978, to December 31, 1979. In addition, we request that Appendix B to the Agreement be replaced by the attached proposed "Appendix B to Agreement between Mobil Chemical Company and Vertac, Incorporated - Revision of ~~November~~ January 1, 1976", which is attached hereto as Exhibit 1.

The purpose of our proposal is to provide a more mutually acceptable long term basis under which our Eagle River Chemical Corporation will manufacture NBE for Mobil. The mutual interests served by this proposal include the considerations discussed below.

Since the contract was entered into on September 1, 1975, Eagle River has demonstrated a high degree of competence in producing the product (NBE) and has at all times performed in a timely and efficient manner. It is a matter of record that the material efficiency factors, since the start-up of the facility, have exceeded original expectations, resulting in a savings which has inured solely to the benefit of Mobil.

It is also recognized that in addition to providing Mobil with a reliable source of NBE, Eagle River has another distinct advantage, namely, it has the ability to add incremental capacity, to meet Mobil's growing requirements for NBE, with modest capital expenditures. Attached hereto as Exhibit 2 is a summary of Eagle River's expansion capability on a three phase program which would raise the plant capacity incrementally from approximately 6,000,000

pounds annually to a total of 12,000,000 pounds annually. The three phases which would raise the annual capacity to 7, 9 and 12 million pounds are presently estimated to cost $50,000; $150,000 and $250,000 respectively. Thus, for a capital expansion totaling $450,000 an additional 6,000,000 pounds of NBE capacity would be provided.

The purpose for proposing a revision to the Agreement at this time is that it will be impossible for Eagle River to continue as a supplier of NBE, on either a short or long term basis, unless provision is made to increase Eagle River's compensation for its manufacturing services recognizing that (1) operating costs have escalated since September, 1975, (2) EXPERIENCE HAS PROVEN THE ORIGINAL EAGLE RIVER COST ESTIMATES FOR THE PRODUCTION OF NBE BY MOBIL'S PROCESS WERE TOO LOW PARTICULARILY IN THE CONSUMPTION OF UTILITIES AND OPERATING SUPPLIES AND IN ITS REQUIREMENTS FOR LABORATORY AND MAINTENANCE SERVICES AND (3) THE FACT THAT THROUGHOUT THE HISTORY OF THE CONTRACT MOBIL HAS BEEN AN UNRELIABLE SUPPLIER OF CBE, THE NBE PRECURSOR, AND DURING PERIODS OF CBE SHORTAGE OR UNAVAILABLILITY THE BASIC COSTS OF THE EAGLE RIVER'S NBE FACILITY HAVE NECESSARILY CONTINUED WITH LITTLE OR NO NBE OUTPUT TO ABSORB THESE COSTS.

ATTACHED HERETO AS EXHIBIT 3 IS A SUMMARY OF OPERATING COSTS AT MONTHLY NBE OUTPUTS OF 400,000 AND 450,000 500,000 POUNDS WHICH PROVIDES THE BASIS FOR OUR REQUEST THAT EAGLE RIVER'S FEE BE INCREASED FROM 16 CENTS PER POUND TO 17 ✓ CENTS PER POUND FOR THE REVISED AGREEMENT SUBJECT TO QUARTERLY ESCALATION OR DEESCALATION AS NECESSARY AND AS PROVIDED IN THE PROPOSED FORMULA.

THE REQUESTED FEE IS BASED ON GOOD FAITH THAT MOBIL WILL PROVIDE, IN THE FUTURE, SUFFICIENT CBE TO PERMIT EAGLE RIVER TO PRODUCE AND TRANSFER TO MOBIL AN AVERAGE OF 450,000 POUNDS OF NBE PER MONTH AT WHICH OUTPUT EAGLE RIVER WOULD EARN AN AFTER TAX RETURN ON THE FIXED INVESTMENT OF 11.5%. AFTER TAX RETURN ON 400,000 POUNDS MONTHLY PRODUCTION WOULD BE 4.7% AND ON 500,000 POUNDS IT WOULD BE 19%. Eagle River's

WE BELIEVE MOBIL WILL RECOGNIZE ~~OUR~~ NEED TO EARN SUCH A PROFIT ON ~~OUR~~ IT'S INVESTMENT AND THAT OUR FULL DISCLOSURE OF ~~OUR~~ KDS OUR OPERATING COSTS WILL SATISFY MOBIL THAT OUR PROPOSED FEE OF $0.17 PER POUND OF NBE IS ESSENTIAL IN ORDER FOR EAGLE RIVER TO CONTINUE AS A RELIABLE SUPPLIER OF NBE.

AS NOTED ABOVE OUR PRICE IS ESTABLISHED ON THE BASIS OF CONTINUOUS PRODUCTION AT A VOLUME BETWEEN 400,000 AND 500,000 POUNDS PER MONTH.

WE WISH TO EMPHASIZE THAT OVER 80% OF OUR COSTS ARE CONSTANT WHETHER OR NOT WE ARE PRODUCING NBE. WHEN THE FACILITY IS DOWN FOR LACK OF RAW MATERIAL ALL FIXED COSTS AND THE BULK OF OUR DIRECT VARIABLE COSTS CONTINUE SINCE WE CANNOT MAINTAIN RELIABILITY BY DISMISSING THE ASSIGNED LABOR FORCE WHILE AWAITING RAW MATERIAL. THE ONLY COSTS

NOT INCURRED DURING SHUTDOWNS FOR MATERIALS ARE A PORTION OF OUR UTILITIES BUT EVEN THESE ARE NOT TOTALLY ELIMINATED.

SINCE OUR QUOTATION PROVIDES ONLY MODEST PROFIT WHILE PRODUCING AT THE MAXIUMUM ANTICIPATED RATES WE MUST HAVE PROTECTION FROM MOBIL FOR STAND-BY COSTS INCURRED BY EAGLE RIVER WHEN AND IF IT IS NECESSARY TO TEMPORARILY SUSPEND OPERATIONS DUE TO FAILURE TO RECEIVE RAW MATERIAL FROM MOBIL. ACCORDINGLY, WE PROPOSE THAT IN ANY CALENDER MONTH THAT NBE PRODUCTION DROPS BELOW 400,000 POUNDS DUE TO LACK OF RAW MATERIALS THAT MOBIL PAY TO EAGLE RIVER A STAND-BY CHARGE OF $75.00 PER HOUR FOR ALL HOURS OUR REACTOR IS DOWN AWAITING RAW MATERIALS IN EXCESS OF 50 HOURS. STAND-BY COST WOULD REINBURSE US FOR OUR OUT OF POCKET COSTS AND FIXED CHARGES BUT WOULD, DURING SUCH STAND-BY PERIODS, DEPRIVE US OF THE PROFITS WE WOULD NORMALLY EXPECT TO EARN ON OUR FIXED INVESTMENT.

EXHIBIT 1

Reg 173

APPENDIX B

TO AGREEMENT BETWEEN

MOBIL CHEMICAL COMPANY

AND

VERTAC, INCORPORATED

REVISION OF JANUARY 1, 1977

1. CONTRACTORS COMPENSATION FOR NBE SHALL BE AS FOLLOWS:

A. FOR ALL NBE DELIVERED HEREUNDER: $0.17 PER POUND.

B. CONTRACTOR SHALL INVOICE MOBIL FOR EACH SHIPMENT OF NBE MADE ON AN INDIVIDUAL SHIPMENT BASIS. PRICES ARE F.O.B. WEST HELENA, ARKANSAS.

C. THE PRICE PER POUND AS PROVIDED FOR IN SECTION 1.A ABOVE SHALL BE SUBJECT TO ESCALATION OR DEESCALATION COMMENCING IN THE SECOND CALANDER QUARTER OF 1977 AND IN EACH SUBSEQUENT CALANDER QUARTER FOR THE LIFE OF THIS AGREEMNT, PROVIDING EAGLE RIVER ADVISES MOBIL 30 DAYS BEFORE THE FIRST DAY OF THE SUBSEQUEST QUARTER THAT A PRICE ADJUSTMENT IS REQUIRED AS A RESULT OF THE FOLLOWING COMPUTATION:

$$\text{FEE} = 0.17\left[0.5\,\frac{LDR^2}{5.63} + 0.35\,\frac{CPI^2}{172.6} + 0.15\left(0.7\,\frac{KWH^2}{0.029} + 0.3\,\frac{MCF^2}{0.81}\right)\right]$$

Look-up

WHERE,

1. 5.63 IS THE AVERAGE LEAD OPERATORS RATE IN $/HOUR FOR THE FIRST QUARTER OF 1977 AND LDR^2 IS THE AVERAGE RATE TO BE PAID IN THE SUBSEQUEST QUARTER.

2. 172.6 IS THE CONSUMER PRICE INDEX FOR SEPTMEBER, 1976. CPI^2 WILL BE THE LAST PUBLISHED CPI FIGURE ABAILABLE BEFORE COMPUTING THE ADJUSTED FEE.

3. 0.029 IS THE COST OF ELECTRICITY in dollars PER KWH ANTICIPATED FOR THE 1ST QUARTER OF 1976. KWH^2 IS THE COST OF ELECTRICITY PER KWH FOR THE LAST BILLING PRIOR TO COMPUTING THE ADJUSTED FEE.

4. 0.81 IS THE COST OF GAS in dollars PER MILLION STANDARD CUBIC FEET ANTICIPATED FOR THE FIRST QUARTER OF 1977. MCF^2 IS THE COST OF GAS PER MILLION STANDARD CUBIC FEET FOR THE LAST BILLING PRIOR TO COMPUTING THE ADJUSTED FEE.

2. IN RECONGNITION THAT EAGLE RIVER IS TOTALLY DEPENDENT UPON MOBIL FOR RAW MATERIAL, PARTICULARLY CBE, AND THAT MOBIL DESIRES EAGLE RIVER TO CONTINUOUSLY MAINTAIN ITS MANUFACTURING CAPABILITY FOR NBE BY RETAINING ITS WORK FORCE IN STAND-BY WHILE AWAITING RAW MATERIAL AND FURTHER MOBIL RECOGNIZES THAT ESSENTIALLY ALL OTHER COSTS OF MANUFACTURING NBE WITH THE EXCEPTION OF CERTAIN UTILITIES CONTINUES WHETHER OR NOT RAW MATERIALS ARE AVAILABLE IT THEREFORE AGREES TO PAY EAGLE RIVER STAND-BY CHARGES TO BE COMPUTED AS FOLLOWS:

IN ANY CALENDER MONTH IN WHICH THE PRODUCTION OF NBE IS LESS THAN 400,000 POUNDS MOBIL WILL PAY TO EAGLE RIVER A STAND-BY FEE OF SEVENTY-FIVE DOLLARS ($75.00)PER HOUR FOR ALL HOURS FOR WHICH PRODUCTION IS INTERRUPTED FOR LACK OF RAW MATERIALS IN EXCESS OF FIFTY (50) HOURS IN SAID MONTH. INTERRUPTION SHALL BE ESTABLISHED FROM THE PLANT LOG WHERE THE ENTRY IS MADE AND WITNESSED BY THE SHIFT SUPERVISOR THAT THE REACTOR IS READY FOR LOADING AND AWAITING THE ARRIVAL OF RAW MATERIALS NEEDED FOR THE FIRST MANUFACTURING STEP. TIME OF ARRIVAL AND AVAILABILITY FOR LOADING SHALL BE NOTED AND WITNESSED BY THE SHIFT SUPERVISOR,

Exhibit, Pg 373

THE FOLLOWING ILLUSTRATIVE CALCULATION DEMONSTRATES THE MANNER IN WHICH, THE STAND-BY FEE SHALL BE COMPUTED.

MONTH - APRIL 1977.

NBE PRODUCED 368,000 POUNDS.

HOURS DOWN TIME DUE TO LACK OF RAW MATERIALS - 72

72-50 = 22 HOURS STAND-BY CHARGEABLE TO MOBIL

22 X $75 = $1650

Exhibit 2
Page 1 of 2

TABLE I

MOBIL NITRATION (NBE) EXPANSION

LEVEL OF EXPANSION	MONTHLY CAPACITY MLBS	ANNUAL CAPACITY MMLBS	CAPITAL * REQ'D $M	ADDITIONAL** EXPENSES	REMARKS
CURRENT	450	+5	none	none	Unit requires shakedown at higher production levels (sustained). Batch cycle times well documented.
I CURRENT ATTAINABLE	600	+7	[redacted]50	add operator on days for tank trucks	Replace selected CS piping and pumps with SS items. Requires slight increase in batch siz and high stream factor. pH control of NH_3 & EDC storage recommended but not essential.
II	800	+9	150-200	add operator on swing shift for tank trucks	Requires additional 3.5M gal SS reactor to allow separate phase separation & neutralization thus freeing nitration reactor. Add additional CBE storage. Add second cooling tower.
III	1000	12	250-450	add third operator spot add day production supervisor for Mobil unit	Requires conversion of 3.5 M gal SS reactor (per above) to nitration service including two acid meas. bottles & their pumps, weigh cell & reactor interlocks, heat exchanger for reactor cooling etc.. Add NBE storage tank, enlarge spent acid & am. sulfate storage all to 20M gal. Add building structure & roof annex for reactor. Add second refrigeration unit. Convert NBE stora (current) to crude NBE/EDC surge. Feed stripper semi-continous "ala" Mobil NBE process from this converted tank.

* Low capital range value represents minimum capital for that phase only. High capital range value represents total capital thru each phase (i.e. total capital thru Phase III is $450M).

** Represents 9 operators Phase I, 10 operators Phase II, & 12 operators Phase III.

TABLE 11
MOBIL EXPANSION

	INSTALLED COST	REMARKS
1. pH control for NH_3 addition	$ 5M	Uniloc type pH probe & transmitter/controller & control valve/SS tubing with valves, flush points, strainer, etc.
2. EDC storage 20M gal CS with pump	20M	Tank +$6M plus pump $1M, concrete foundation, piping, level instrumentation cons. vent & flame arrestor.
3. Piping & Equipment revisions	25M	Replace CS process pumps with SS. Replace CS piping in EDC system, stripper vent, system etc.
TOTAL	50M Phase I	
4. Add reactor 3.5 M SS with transfer pump	75M	Base reactor $20-30M per J. E. Fowler, Times 3 for foundation, aqua NH_3 & process piping, electrical etc. Include weigh cell & base structural.
5. CBE storage 20M gal CS with pumps	25M	Ditto above item 2 plus cost of panelcoil (STM heated) & insulation.
6. Cooling tower	50M	Installed cost plus pumps by new or good shape used Marley Model 453-201 or similar ($24M~1/30/75).
TOTAL	150M Phase 2	
7. Acid feed tanks & pumps	20M	Two small 1Mgal CS (H_2SO_4) & aluminum (HNO_3) acid feed tanks with small (ECO) metering pumps ($5M times 4)
8. Enlarge Spent Acid & Am. sulfate storage	15M	Add CS rings to go from 10 Mgal to 20M gal in the field (Materials $7M & labor $8M)
9. Reactor Heat Exchanger & large circulating pumps	40M	Heat Exchanger (10M), pump $6M Bare Cost
10. NBE Storage 20Mgal CS with pump	25M	Ditto 5
11. Building, Structure & roof	35M	Structural steel for additional large bay & roof (transite) to house new reactor, acid feed system reactor heat exchanger & refrigeration.
12. Refrigeration	100M	150M ton new or good used (J.E. Fowler $30M similar unit prod. 21—$65 new unit 1/30/75 quote).
13. Modify stripper for semi-continous operation & other minor changes	15M	Revise piping, add level instrument or weigh cell to stripper.
TOTAL	250M Phase 3	
TOTAL EXPANSION	$450M	

VERTAC
INCORPORATED
Building 4—Suite 412 • Shreve City Office Park • Shreveport, Louisiana 71105 • Phone 318-869-3434

July 17, 1975

Mr. Don Faggert
Mobil Chemical Co.
Phosphorus Division - Crop Chemicals Group
P. O. Box 26683
Richmond, Virginia 23261

Subject: NBE Technical Information

Dear Don:

As we discussed, the people at Eagle River need the maximum technical information you can provide in order to build the best nitration unit in the shortest possible time. The following is a list of documents, realizing that all of this may not be available:

One (1) set engineering flow sheets - Mt. Pleasant, Tenn. nitration.
One (1) set process flow sheets.
NBE Analytical Manual with sample schedule, typical analyses and analytical methods.
Physical Properties Data (NBE and raw materials).
One (1) set operating data sheets.
Summary of analyses from past production runs.
Operating Manual.
Detailed equipment lists.
Summary of process description.
Description of principal effluent streams.
Technical Data Bulletin - Safety Data Sheets.
Raw and Process Material Yield Summary.
Product Specification Sheet.

Please send directly to Eagle River Chemical Company, P. O. Box 2648, West Helena, Arkansas 72390, Attention of George Mather.

As I mentioned to you, George Mather and Jim Fowler would like to visit the Mt. Pleasant facilities at a later date.

Best regards,

Bob Fabian

R. M. Fabian
Manager, Business Develop.

RMF-el

Mather

VERTAC
INCORPORATED
Building 4—Suite 412 • Shreve City Office Park • Shreveport, Louisiana 71105 • Phone 318-869-3434

July 8, 1975

Mr. Don Faggert
Mobil Chemical Company
Phosphorus Division - Crop Chemicals Group
P. O. Box 26683
Richmond, Virginia 23261

Subject: Proposal for CBE Nitration

Dear Don:

Confirming our telecon, the following toll charge basis is mutually agreed upon:

First 2MM lbs.	-	19¢ per lb.
Subsequent production	-	16¢ per lb.

To compliment the above, you agreed to the following:

1) Pre-payment of $200,000 to be applied against initial production @ 19¢ per lb. rate.
2) Firm "take-or-pay" for 5MM lbs.
3) Production schedule as follows to be included in the contract:

    a) First 6 months - 2MM lbs.
    b) 500,000 lbs/mo thru January, 1978

You stated that your lawyers would be reviewing the contract draft on July 10th. If it appears there will be a delay of a week or more, please send a letter of intent by July 15, 1975. This is a must if we are to meet the following schedule:

October 1, 1975	- Mechanical completion
October 15, 1975	- Plant Start-up

Continued . . . . . .

Don, we are pleased that Mobil and Vertac have reached this agreement. I'm sure it will prove to be beneficial to both parties.

Best regards,

R. M. Fabian
Business Development Manager

RMF-el

cc: N. D. Morgan
R. A. Guidi
J. W. Shackelford
J. Miles
G. Mather

Both John - File
Send copy to Matthew
Attachment #1 only

VERTAC
INCORPORATED
Building 4 — Suite 412 • Shreve City Office Park • Shreveport, Louisiana 71105 • Phone 318-869-3434

July 1, 1975

Mr. Don Faggert
Mobil Chemical Co.
Phosphorus Division - Crop Chemicals Group
P. O. Box 26683
Richmond, Virginia 23261

Subject: Proposal for CBE Nitration

Dear Don:

The attachments present all the background data we have used to determine our toll charge proposal to your company. The details supporting the schedule shown below are provided in attachment #1.

Monthly Production	Toll Charge
100-149 M#/mo	.37¢
150-199 "	.32¢
200-249 "	.27¢
250-299 "	.24¢
300-349 "	.21¢
350-399	.19¢
400-449	.17.5¢
450-500	.16¢

John Miles' work-up is included as attachment #2.

Our principal concerns are adequate pay-back on $200,000 capital and sufficient profit at reduced production levels to make it worth our effort. Also, considering the very high dollar value of these chemicals, we expect Mobil to retain title thru-out the manufacturing operation.

Continued.....

Don, I'm sure we are getting close to reaching agreement on a toll charge schedule. You said Mobil will send us a proposed contract format by July 7th. To insure meeting an October 1st start-up date we must have a letter of intent in our hands by July 15, 1975. If I can be of further assistance, please call me.

Best regards,

Bob Fabian

R. M. Fabian
Business Development Manager

RMF-el

cc: Ray Guidi (Attachment #1)
N. D. Morgan (Attachment #1)
John Miles - Eagle River (Attachment #1)

John Miles

VERTAC
INCORPORATED
Building 4—Suite 412 • Shreve City Office Park • Shreveport, Louisiana 71105 • Phone 318-869-3434

July 1, 1975

Mr. Don Faggert
Mobil Chemical Co.
Phosphorus Division - Crop Chemicals Group
P. O. Box 26683
Richmond, Virginia 23261

Subject: Proposal for CBE Nitration

Dear Don:

The attachments present all the background data we have used to determine our toll charge proposal to your company. The details supporting the schedule shown below are provided in attachment #1.

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John Miles' work-up is included as attachment #2.

Our principal concerns are adequate pay-back on $200,000 capital and sufficient profit at reduced production levels to make it worth our effort. Also, considering the very high dollar value of these chemicals, we expect Mobil to retain title thru-out the manufacturing operation.

Continued.....

ATTACHMENT #1

OUTLINE OF VERTAC POSITION FOR MOBIL NITRATION

PROPOSAL:

1. $200M capital is required to achieve 330M #/mo. rate applicable during 1st 6 mos.; this "front-end" money must be pre-paid.

2. Total investment required for Mobil needs @ 500M #/mo. is $350M; we must recover $200M of this within 1-2 years (after taxes); also, "take or pay" arrangement must insure that we at least recover this amount.

3. We must be covered for our actual manufacturing cost incurred regardless of monthly production rate - see attached estimated manufacturing costs for various production levels.

4. Toll charge (Manufacturing cost + profit) must be adequate to pay-back $200M investment within 1-2 years regardless of monthly production rate:

Monthly Capacity	Mfg. Costs	Profit	Toll Charge
100M#/mo	.27¢	.12¢	.39¢
250M#/mo	.15¢	.10¢	.25¢
330M#/mo	.12¢	.09¢	.21¢
500M#/mo	.08¢	.08¢	.16¢

(See plot for actual production brackets.)

5. The following production schedules would insure items #2 & #4 above (take 18 mos. period):

a) Assume first 6 mos. @ 2.0$\overline{M}$ lbs/mo & subsequent 12 mos. @ 500M lbs/mo ea.
B.T. Profit = 2.0$\overline{M}$ ($.09) + [12 x 0.5$\overline{M}$ ($.08)]
= $180M + $480M = $660M
A.T. Profit = $330M (OK)

b) Assume first 6 mos. @ 2.0$\overline{M}$ lbs/mo. & subsequent 12 mos. @ 250M lbs/mo. ea.
B.T. Profit = $180M + [12 x .25 $\overline{M}$ (.10)]
= $180M + $300M = $480M
A.T. Profit = $240M (OK)

c) Assume first 6 mos. @ 2.0$\overline{M}$ lbs/mo & subsequent 12 mos. @ 100M lbs/mo. ea.
B.T. Profit = $180M + [12 x 100M (.12)]
= $180M + $144M = $324M
A.T. Profit = $162M (somewhat short)

Note: $\overline{M}$ = Million
M = Thousand

What is pay-back period?

Monthly gross profit A.T. = $6M

Additional mos. to pay-back $= \frac{(200M - 90M)}{6M}$

$= 18.3$

Total pay-back period = 6 + 18.3 = 24 + mos. (Acceptable)

d) If a total cash flow (in excess of contractual manufacturing cost) of $400M has not occurred to Vertac within 24 mos. of initial product delivery, Mobil shall make up the difference and Vertac has the option to cancel the contract.

6. The toll charge of 21¢ shall apply during the first 3 mos. of the contract; manufacturing cost for billing purposes subsequently will be determined quarterly based on actual average monthly shipments for preceding three mos. Since a graph such as attached is not practical for accounting use, production brackets of 50M #/mo. increments could be established.

7. Mobil will retain ownership of all chemicals used in the manufacture of this product.

a) Vertac will provide raw material consumptions on monthly basis.
b) Vertac will notify Mobil within 48 hrs. of any unusual loss, spill, etc..
c) Vertac will bill Mobil directly for any acids, EDC, or ammonia (either anhydrous or aqua) which Vertac purchases.
d) Both parties will examine yields every 90 days and adjust standards per mutual agreement.

ESTIMATED MANFACTURING COST
FOR ADDED PRODUCTION, ANNUAL BASIS DACO-613A

APPROPRIATION NUMBER RMF 6-30-75

Mobil Nitration Proposal

		* Fixed Cost @ Full Rate Reduced 1/2	Full Fixed Cost	Full Fixed Cost
1.	PLANT PRODUCTION			
	RATE, ____ OF ____ / ____			
	RATE, IN % OF NEW TOTAL CAPACITY			
	ADDED, IN ____ OF ____ / 165/mo. →	100,000	250,000	500,000
	ADDED, IN % OF ADDED CAPACITY	20 %	50 %	100 %
2.	RAW MATERIAL & FUEL COST			
	TYPE / UNITS / UNIT COST / USAGE / ____	$	$	$
	SUBTOTAL, RAW MATERIALS & FUEL	$ —	$ —	$ —
3.	UTILITIES			
	TYPE / UNITS / UNIT COST / USAGE / ____	$	$	$
	ELECTRICITY 600 kwh @ 2.5¢/kwh			
	STEAM 17,000# @ ¢/- /m #			
	WATER 12,000 gal @ 10¢/m gal			
	Refrig. 1.5x10⁶ Btu @ .033¢/m Btu			
	SUBTOTAL, UTILITIES	$ .008	$ .008	$ .008
* 4.	OPERATING LABOR $ 2 men/shift	$ .043	$ .035	$ .017
5.	ROYALITIES & RENTALS $ ____ / ____	—	$ —	$ —
6.	BURDEN			
	A. REPAIR LABOR	$ .028	$ .022	$ .011
*	B. REPAIR SUPPLIES (incl. O.H.)			
	C. OPERATING SUPPLIES	.065	.026	.013
	D. SUPERVISION — #135			
	E. INDIRECT PAYROLL @ ____ Included above	—	—	—
	SUBTOTAL, BURDEN	$ .093	$ .048	$ .024
7.	FIXED AND SERVICE COSTS			
*	A. GENERAL WORKS EXPENSE, INCL. TAXES & INS. #85.62	$ .020	$ .016	$ .008
	B. DEPRECIATION #3.50M — 5 yr. S.L. — #74.91	.035	.014	.007
*	C. LABORATORY — #26/month	.005	.004	.002
	D. Interest #71.26	.035	.014	.007
	SUBTOTAL, FIXED & SERVICE COSTS	$ .095	$ .048	$ .024
8.	TOTAL BULK COST	$ .239	$ .139	$ .073
9.	LOADING, PACKING & SHIPPING			
	A. ____			
	B. ____			
	SUBTOTAL, L.P. & S.	$	$	$
10.	CONTINGENCIES @ 10% #77.73	$ .028	$ .014	$ .007
11.	TOTAL MFG. COST FOR ADDED PROD. ANNUAL BASIS	$ .267	$ .153	$ .08
12.	UNIT MANUFACTURING COST ~~FOR ADDED PRODUCTION~~	$ .120	$ .100	$ .08
	Tolling Chg. →			
	Total Prod. Cost.	.387	.253	.16

NOTES:

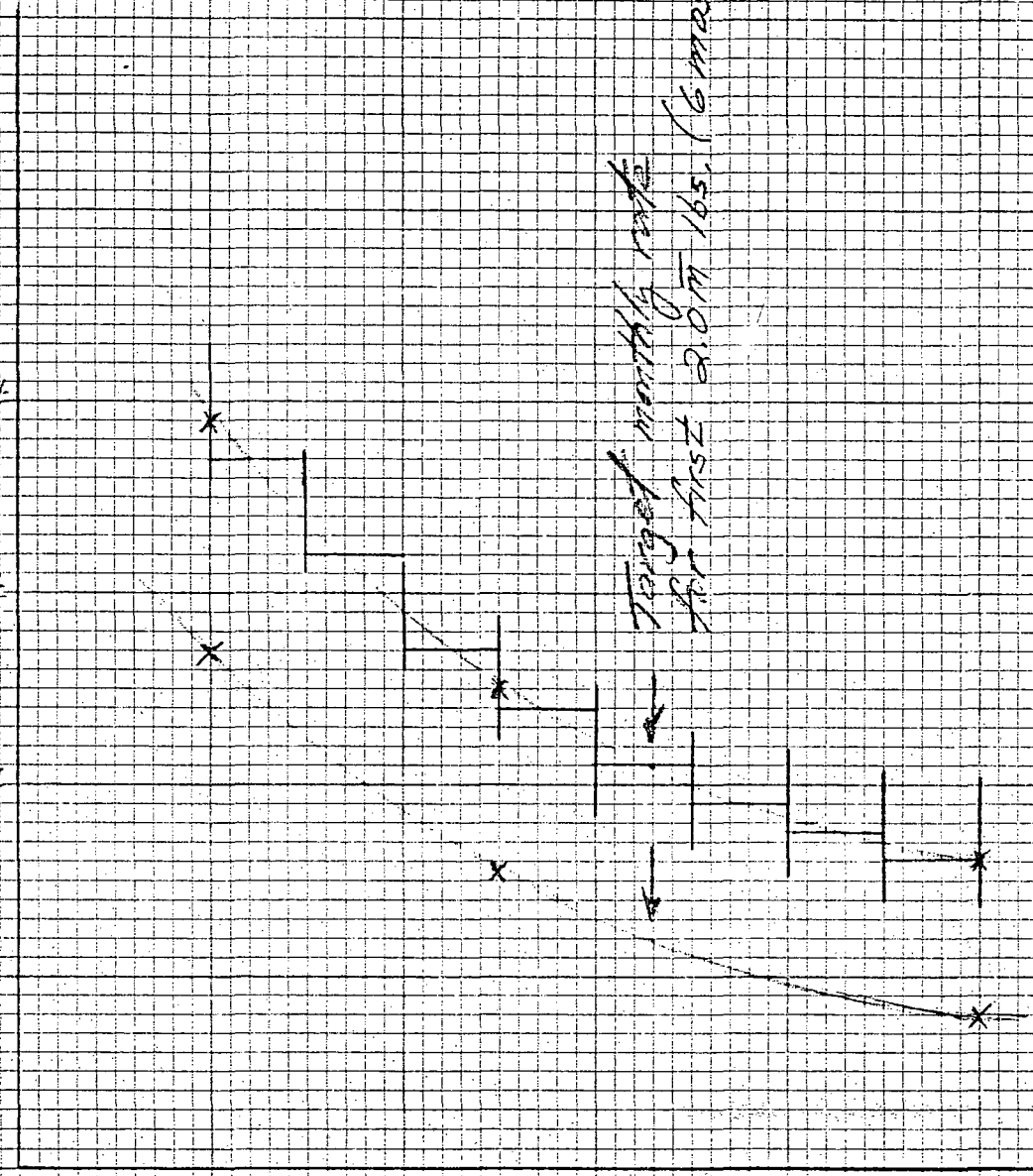
Note: To get cost per lbs, John Miles batch cost figures were divided by 10,417 lbs per batch @ 500M lbs/mo.; Batch size for 250M lbs/mo. is one-half of above.

PREPARED BY ____ DATE ____ APPROVED BY ____ DATE ____

Mobil Nitration Proposal

R. Fabian 6/30/75

Monthly Capacity M#	Total Toll Chg.
100-149	37¢
150-199	32¢
200-249	27¢
250-299	24¢
300-349	21¢
350-399	19¢
400-499	17.5¢
450-500	16¢



Don, I'm sure we are getting close to reaching agreement on a toll charge schedule. You said Mobil will send us a proposed contract format by July 7th. To insure meeting an October 1st start-up date we must have a letter of intent in our hands by July 15, 1975. If I can be of further assistance, please call me.

Best regards,

Bob Fabian

R. M. Fabian
Business Development Manager

RMF-el

cc: Ray Guidi (Attachment #1)
N. D. Morgan (Attachment #1)
John Miles - Eagle River (Attachment #1)

John Niles

INTER-OFFICE CORRESPONDENCE

VERTAC
INCORPORATED
Suite 3200 — Clark Tower
5100 Poplar Avenue
Memphis, Tennessee 38137

June 18, 1975

TO: Niven Morgan
Larry Wallace
Bill Shackelford

FROM: Larry Graybill

SUBJECT: Revisions for letter to Don Faggert dated June 13, 1975.

REPLY REQUESTED BY (DATE)

Attached is a copy of page 3 of the letter to Don Faggert dated June 13, 1975 with revisions for the Operating labor, Miscellaneous operating expense, and Total figures.

If you have any question, please call.

Sincerely,

Larry (lc)

Larry Graybill

LLG/bc
ENCLOSURE

Blind note: As a review, the plans are to manufacture the first six months requirements utilizing the present DCNB production train and then install a larger 3,000 gallon stainless steel reactor, which will probably be necessary to achieve the higher production rate. The total cost of the project is estimated at $ 350,000.00 and is outlined in detail in my May 28 review of the project to Niven Morgan. We feel quite comfortable that the 350 number is more than adequate to accomplish what we are proposing to do. The 32¢ proposed charge to Mobil gives us a very attractive return and will have some room for negotiation if necessary. The charge broken down is as follows:

	DOLLARS PER POUND
Raw materials	$ -0-
Utilities	.0081
Operating labor	.0173
Maintenance	.0108
Miscellaneous operating expense	.0162
General works overhead	.0082
Depreciation	.0072
Interest	.0068
Contingency	.0075
Gross profit	.2379
Total	$ .3200

These figures are based on 500,000 lb./month operating rate.

This is a new product for Mobil and has the normal uncertainties regarding long-term volume. Therefore, the termination option will allow either party to walk away from the contract if it is bad fit. I personally recommend this approach rather than trying to cover all avenues and cross all bridges in a formal contract. The firm commitment on Mobil's part for 2,000,000 lbs. of product should give us adequate protection in paying for the multi-purpose plant that we are putting out. I also think this simple conract approach will be more palatable to Mobil an easier to get a quick resolution and signing.

John Miles

VERTAC INCORPORATED Suite 2900—Clark Tower • 5100 Poplar Avenue • Memphis, Tennessee 38137 • 901-761-0050

June 13, 1975

Mr. Don Faggert
Mobil Chemical Company
P. O. Box 26683
Richmond, VA 23261

Dear Don:

Based on our several meetings, the technical exchange in Richmond and the secrecy aggreement signed February 28, 1975, we are submitting a formal proposal for the manufacture of NBE (2 chloro-5 nitromethyl-benzoate) from CBE (meta chloromethylbenzoate). Based on Mobil's production of this product in their own plant facilities and your review of our engineering flow sheet submitted by John Miles on June 4, 1975, we are in agreement that sufficient know-how exist between our companies for Eagle River Chemical to install and operate the manufacturing facilities.

Per our last conversation, we would like to have a decision firmed by late June so that we can proceed on the revamping of our facilities to manufacture your product. If we are in agreement on the project, we should be able to proceed on a letter of intent on Mobil's part while we are working out the contract details. However, as we discussed hopefully we can firm up the contract arrangements by using an existing formulation type contract that you currently have with Helena Chemical Company. Towards that end I have enclosed copies of Helena's standard contract manufacturing agreement as well as a recent contract signed with that group by Mobil. I would suggest that we use these existing formulation contract formats and incorporate the following items:

(1) Contract production amounts - 13,000,000 lbs. of NBE supplied as follows: 1,600,000 lbs. to 2,000,000 lbs. supplied during the first six-month period commencing October 1, 1975. Thereafter, 400,000 to 500,000 lbs. per month through January, 1978.

(2) Mobil will supply all raw materials and will retain title during the tolling process. Mobil will also be responsible for the disposal of all waste effluents from the process.

$160,000

(3) Eagle River's charge for this tolling operation will be 32¢/lb., F.O.B. Mobil tankcar at Eagle River's plant sight in West Helena, Arkansas. To off-set the revamping charges to modify the plant Mobil will prepay for 500,000 lbs. of product on plant start-up.

(4) Mobil will supply tankcars for incoming raw material storage and

shipment of finished products.

(5) Mobil and Eagle River will mutually establish raw material yield rates after the first ninety (90) days of normal plant operation. If Mobil is not satisfied with the yields achieved, or the parties cannot agree to normal yield rates, Mobil has the right to assume direct supervision of the plant process to determine the proper yields.

4 640,000 TOTAL
164,200 O.C.
475,800 - TO COVER INVESTMENT

(6) Either party can terminate the agreement with sixty (60) days notice after the production of the first 2,000,000 lbs. of finished product. The first 2,000,000 lbs. will be on firm take or pay basis.

(7) The contract should outline reasonable order placement procedures and invoicing at shipment on net thirty (30) day terms.

(8) The contract should include formal specifications on all raw materials and finished products.

(9) The contract should include normal force majeure protection for both parties.

Don, I think that this summarizes our agreement to date. If you have additional points or changes, please let me know; and if your legal department will contact our council, Larry Wallace (address: Wallace, Hilburn & Wilson, LTD., Attorneys-at-Law, 219 Main, North Little Rock, Arkansas 72114, phone: (501)376-9993), prior to the drafting of the contract, I think we will save some time.

Please let your people know that we certainly appreciate the opportunity to quote on this project and are pleased with the exchanges that have taken place to date. I will be in touch with you shortly to setup a meeting date in Richmond to introduce my replacement to you and your people there.

Sincerely,

Larry Graybill
Manager
Business Development

LLG/bc
cc: Niven Morgan
Larry Wallace
Bill Shackelford

Blind note: As a review, the plans are to manufacture the first six months requirements utilizing the present DCNB production train and then install a larger 3,000 gallon stainless steel reactor, which will probably be necessary to achieve the higher production rate. The total cost of the project is estimated at $ 350,000.00 and is outlined in detail in my May 28 review of the project to Niven Morgan. We feel quite comfortable that the 350 number is more than adequate to accomplish what we are proposing to do. The 32¢ proposed charge to Mobil gives us a very attractive return and will have some room for negotiation if necessary. The charge broken down is as follows:

		DOLLARS PER POUND	
Raw materials	$	-0-	
Utilities		.0081	.00811
Operating labor		.0873	.01728
Maintenance		.0108	.01075
Miscellaneous operating expense		.0862	.01622
General works overhead		.0082	.00822
Depreciation		.0072	.00719
Interest		.0068	.00684
Contingency		.0075	.00746
Gross profit		.2379	.23793
Total	$	.4600	.32000

.08207

These figures are based on 500,000 lb./month operating rate.

This is a new product for Mobil and has the normal uncertainties regarding long-term volume. Therefore, the termination option will allow either party to walk away from the contract if it is bad fit. I personally recommend this approach rather than trying to cover all avenues and cross all bridges in a formal contract. The firm commitment on Mobil's part for 2,000,000 lbs. of product should give us adequate protection in paying for the multi-purpose plant that we are putting out. I also think this simple conract approach will be more palatable to Mobil an easier to get a quick resolution and signing.

Note to the File.

Mobil Nitration Meeting.

May 20, 1975, Richmond, VA.

Meeting attended by Larry Graybill
Larry Conaway - Vertac
John Miles

Don Faggert, Contract Manufacturing Manager
John Randle, Manager - Process Engineering, Phosphorous Division
John Ellington, Process Engineer - Mt. Pleasant Plant - Mobil
Julian Wolfenbarger, Process Engineer - Phosphorous Division
Ted Nichols, Process Engineering Manager

Purpose of the meeting was to evaluate the Vertac proposal to convert CBE (meta chloromethylbenzoate) to the NBE (2 chloro-5 nitromethylbenzoate). This was a technical meeting to further define the chemistry and engineering requirements necessary to make a final economic proposal to Mobil by mid-June.

Mobil is starting up a Modown unit at their Mt. Pleasant facilities July 1, 1975. The unit is built around 10,000 gallon reactor which is designed to take the CBE from Velsicol and nitrate into the NBE and then further reacted into the finished Modown product. However, Mobil is interested in our nitrating the CBE so that their unit is devoted entirely to the finished product manufature. Their requirements are as follows:

October, 1975	300,000 Lbs./mo.	
November & December	240,000 Lbs./mo.	
January	370,000 Lbs./mo.	
February & March	430,000 Lbs./mo.	
April, 1976 through January, 1978	500,000 Lbs./mo.	TOTAL: 13,010,000 Lbs.

These NBE requirements are tied to the CBE availability as forecasted by Velsicol for Mobil's use. Velsicol is currently doing the esterfication of the benzoyl chloride. Although not discussed, I would suspect that by January, 1978 Mobil's market would have grown to the point that they could establish their own Modown unit designed for a higher capacity.

Mobil is evaluating a proposal from another contract manufacture for this nitration operation, but I feel that our overall relationship between Mobil and Helena would help us get the nod or inside track. My earlier proposal of manufacturing cost at 13¢/pound and a tolling charge of 24¢/pound was in the ballpark and of interest to Mobil. Based on the information listed below as well as other physical data transmitted to us, we are to revise our engineering plan and re-evaluate the overall economics.

VERTAC
INCORPORATED
Suite 3200 — Clark Tower
5100 Poplar Avenue
Memphis, Tennessee 38137

INTER-OFFICE CORRESPONDENCE

June 4, 1975

TO: Bill Shackelford

FROM: Larry Graybill

SUBJECT: Mobil Visit.

REPLY REQUESTED BY (DATE)

Ted Nicholls, Manager of Project Engineering, and Don Faggert, Contract Manufacturing Manager, will arrive in Memphis on Monday evening, June 9. I will pick them up Tuesday and we should arrive at Eagle River around 9:00 to 9:30.

The purpose of the meeting is threefold: (1) review the preliminary engineering scheme as outlined by John Miles to meet the higher production rate. (2) Acquaint Ted Nicholls with the plant sight and give them the assurances they need regarding our ability to have on stream a nitration unit by October, 1975. (3) We will return to Memphis for a preliminary business meeting to discuss economics and the proposed contract points.

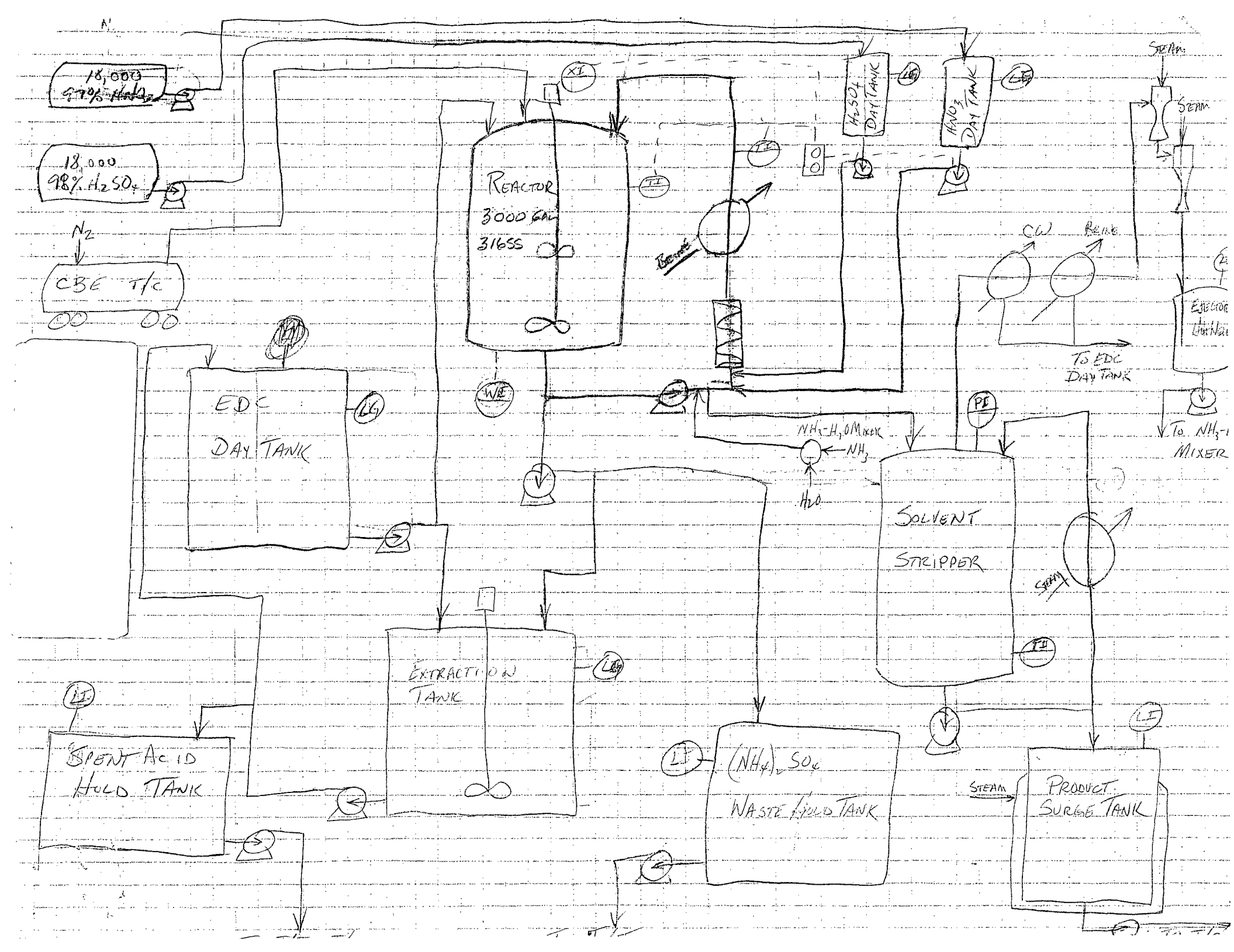
I will be in touch with John Miles prior to the meeting to discuss and refine our position.

Sincerely,

Larry (bc)

Larry Graybill

LLG/bc
cc: Larry Connaway
John Miles
Niven Morgan
Ray Guidi


N2
18,000
98% H2SO4
18,000
98% H2SO4
N2
CBE T/C
REACTOR
3000 GAL
316SS
H2SO4 DAY TANK
HNO3 DAY TANK
STEAM
STEAM
CW
BRINE
TO EDC DAY TANK
EDC
DAY TANK
NH3-H2O MIXER
NH3
H2O
SOLVENT
STRIPPER
STEAM
To NH3-H2O MIXER
EXTRACTION
TANK
SPENT ACID
HOLD TANK
(NH4)2SO4
WASTE HOLD TANK
STEAM
PRODUCT
SURGE TANK

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SPEED LETTER®

TO John Niles ER

FROM **VERTAC, INC.**

MEMPHIS OFFICE

SUBJECT

No. 9 & 10 FOLD

MESSAGE

DATE 5/28 19 75

John, attached is a copy of the report submitted to Niven along with the original of your Notes on the meeting. I would recommend you diary suggest a copy of your notes on the meeting and ask him to correct any errors or omissions on our part.

SIGNED Larry

REPLY

DATE 19

No. 9 FOLD

No. 10 FOLD

SIGNED

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274

SNAP-A-WAY AND RETAIN YELLOW COPY. SEND WHITE AND PINK COPIES WITH CARBON INTACT

Bill Shackelford

VERTAC INCORPORATED Suite 2900—Clark Tower • 5100 Poplar Avenue • Memphis, Tennessee 38137 • 901-761-0050

May 27, 1975

Don Faggert
Manager, Crop Chemicals Formulation
Mobil Chemical Company
P. O. Box 26683
Richmond, VA 23261

Dear Don:

Thanks for the opportunity to present our nitration proposal. We appreciated the open and serious exchange of information supplied by your technical people.

Based on the more complete information supplied in this meeting and your increased production requirements, we are revising our proposal. The preliminary engineering work should be completed this week, and we will be in a position to discuss with you during your next visit to Helena here in Memphis. We should then be able to make a firm proposal during mid-June.

Again, we appreciate the opportunity to work on this project with Mobil and look forward to expanding the good Mobil-Helena relationship to include our Vertac group. Let me know your travel plans, and I will look forward to seeing you then.

Sincerely,

Larry

Larry Graybill
Manager
Business Development

LLG/bc

VERTAC
INCORPORATED
Suite 3200 — Clark Tower
5100 Poplar Avenue
Memphis, Tennessee 38137

INTER-OFFICE CORRESPONDENCE

June 4, 1975

TO: Bill Shackelford

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SUBJECT: Mobil Visit.

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I will be in touch with John Miles prior to the meeting to discuss and refine our position.

Sincerely,

Larry (bc)

Larry Graybill

LLG/bc
cc: Larry Connaway
John Miles
Niven Morgan
Ray Guidi

VERTAC
INCORPORATED
Building 4—Suite 412 • Shreve City Office Park • Shreveport, Louisiana 71105 • Phone 318-869-3434

August 1, 1975

Mr. Don Faggert
Mobil Chemical Company
P. O. Box 26683
Richmond, Virginia 23261

Re: CBE Nitration Contract

Dear Don:

In general, your contract proposal is acceptable and looks quite workable. However, I feel we must negotiate the following items in our best interest:

1. Revise "Failure-to-deliver" penalty dates as follows (ref. p. 6 and App. B - 3 (a)):

   (a) Contractor to ship 75,000 lbs. by December 4, 1975.

   (b) Mobil to receive 250,000 lbs. by January 4, 1976.

   (c) Mobil to receive 500,000 lbs. by January 31, 1976.

   No penalty or contract termination option can be exercised if Contractor meets any two of the above three conditions.

2. Re App. B - 3 (b), this condition must be "two-way" to protect us against sustained low production rates. Accordingly, we need termination right if Mobil hasn't taken 5,000,000 lbs. by March 1, 1977.

3. Amount and timing of "front-end" money are not satisfactory. Based on the revised capital estimate of $350M, I had requested $200M prepayment in my letter dated July 8, 1975 to you. We are a highly leveraged company, and cash flow is a constant concern. Accordingly, we must have the following:

   (a) Prepayment of $100M at time of contract signing (target September 1).

   (b) Additional prepayment of $100M at plant start-up (target November 28).

   (c) Total prepayment to be paid back to Mobil at rate of 5¢/lbs. (unless contract is terminated for cause).

4. The "take-or-pay" buy out provision is much too favorable to Mobil, i.e., you actually pay less the earlier you buy out. We must be protected against an early buy-out at least to the extent of our unit profit plus fixed costs that would continue for some time; this is the basis for the 11¢/lbs. charge.

Don, these are the major items of concern to us. There are other minor points of clarification, etc., that we can discuss at Eagle River on Friday, August 8. I would like to see us get this contract wrapped up no later than September 1.

Best regards,

R. M. Fabian
Business Development Manager

RMF:ct

Enclosure

cc: Mr. N. D. Morgan
Mr. R. A. Guidi

VERTAC
INCORPORATED
Suite 3200 — Clark Tower
5100 Poplar Avenue
Memphis, Tennessee 38137

INTER-OFFICE CORRESPONDENCE

June 18, 1975

TO: Niven Morgan
Larry Wallace
Bill Shackelford ✓

FROM: Larry Graybill

SUBJECT: Revisions for letter to Don Faggert dated June 13, 1975.

REPLY REQUESTED BY (DATE)

Attached is a copy of page 3 of the letter to Don Faggert dated June 13, 1975 with revisions for the Operating labor, Miscellaneous operating expense, and Total figures.

If you have any question, please call.

Sincerely,

Larry (bc)

Larry Graybill

LLG/bc
ENCLOSURE

Blind note: As a review, the plans are to manufacture the first six months requirements utilizing the present DCNB production train and then install a larger 3,000 gallon stainless steel reactor, which will probably be necessary to achieve the higher production rate. The total cost of the project is estimated at $ 350,000.00 and is outlined in detail in my May 28 review of the project to Niven Morgan. We feel quite comfortable that the 350 number is more than adequate to accomplish what we are proposing to do. The 32¢ proposed charge to Mobil gives us a very attractive return and will have some room for negotiation if necessary. The charge broken down is as follows:

	DOLLARS PER POUND
Raw materials	$ -0-
Utilities	.0081
Operating labor	.0173
Maintenance	.0108
Miscellaneous operating expense	.0162
General works overhead	.0082
Depreciation	.0072
Interest	.0068
Contingency	.0075
Gross profit	.2379
Total	$ .3200

These figures are based on 500,000 lb./month operating rate.

This is a new product for Mobil and has the normal uncertainties regarding long-term volume. Therefore, the termination option will allow either party to walk away from the contract if it is bad fit. I personally recommend this approach rather than trying to cover all avenues and cross all bridges in a formal contract. The firm commitment on Mobil's part for 2,000,000 lbs. of product should give us adequate protection in paying for the multi-purpose plant that we are putting out. I also think this simple conract approach will be more palatable to Mobil an easier to get a quick resolution and signing.

8/18/75

SUBJECT: COMMENTS ON MOBIL INFORMATION SENT TO US/ALSO STEVE POCSIKS COMMENTS ON PROJECT

COMMENT	REMARKS
(1) MOBIL REACTANT CHARGES DIFFERENT THAN ORIGINAL DATA.	(1) NO SIGNIFICANT DEVIATIONS. MODIFY MATERIAL BALANCE AT A LATER DATE. MOBIL SHOWS MORE EDC, H_2SO_4 & LES HNO_3 THAN ER DESIGN.
(2) WHAT SHOULD WE DO ABOUT REACTIONS IN (8) LOOP ON R-M101 PRIOR TO EACH Ø SEPARATION?	(2) MOBIL INFO SAYS CLOSE DISCHARGE ON R-M101 AND ''PUMP LINE EMPTY. DON'T BELIEVE THIS WILL WORK. SUGGEST N_2 BLOW CONNECTION.
(3) MOBIL DESIGN USES 25% WT. NH_4OH WHILE ER CALLS FOR 14% WT. NH_4OH	(3) DESPITE GREATER EFFLUENT BELIEVE ER SYSTEM WILL BE MORE EFFICIENT (REQUIRES LESS EQUIPMENT AND LESS NH_3 OUR NH_4OH TANK T-M107 IS NO CAPABLE OF HANDLING >14% NH_4OH. NO CHANGE.
(4) MOBIL INFO SHOWS CRUDE NBE R-M101 TO V-M101 VIA SMALL INTERFACE PUMP.	(4) MODIFY DWGS. TO REFLECT THI CHANGE. R-M101>P-M111>V-M10 (SCRATCH P-M107 TRANSFER LINE)
(5) MOBIL USES AN INTERFACE TANK SYSTEM TO RECOVER HARD TO DETECT INTERFACES.	(5) MOBIL SYSTEM SEEMS PRONE TO ERROR. PHASE PROBLEMS COUL BE DRUMMED IF NECESSARY. NO ACTION.
(6) MOBIL USES AN EDC WASH AFTER THE STRIPPING (V-M101) AND NEUTRALIZATION (R-M101)	(6) EDC FLUSH CONNECTION BETWEE R-M101 AND V-M101 COULD BE USED IN NECESSARY. NO ACTION.
(7) MOBIL EXTRACTOR TANK HAS EXCELLENT MIXING.	(7) T-M104 DOES HAVE 4 BAFFLES TURBINE MIXER. GFM TO ✓ HP INPUT & RPM.
(8) MOBIL INFO NOW CALLS FOR JUST ONE GALLON OF HNO_3 PREADDITION VS 15% OF HNO_3.	(8) EITHER TYPE OF PREADDITION CALLS FOR AN INTERLOCK OVER RIDE. THE ONE GALLON HNO_3 HOWEVER COULD BE SATISFIED BY A TIME DELAY CIRCUIT.
(9) MOBIL INFO PINPOINTS NITROGEN NEEDS, LETDOWN STATIONS, ETC. MOBIL INFO INDICATES EDC, NBE, AND CNE ARE ALL FLAMMABLE ALTHO EDC PARTICULARLY SO (EDC=56°F, NBE=341°F AND CBE=244°F)	(9) PROVIDE TIME DELAY SYSTEM DEFINITION SUGGESTED VIA. SIMPLE UTILITY P&ID. SUGGEST 25 PSIG FOR PRESSURING AND ? INCHES FOR PADDIN EQUIPMENT. ACTION DEFINE NITROGEN SYSTEM.

COMMENTS		REMARKS	
(10)	DITTO ABOVE FOR OTHER UTILITIES. A VERY SIMPLE P&ID IS SUGGESTED. BECAUSE OF SIMPLICITY OF THE PROJECT, A NUMBER OF UTILITIES COULD BE SHOWN ON EACH DWG. SUGGESTED PID FORMAT AS A CRUDE OVERLAY OF PLOT PLAN.		
(11)	MOBIL SYSTEM OF NH_3 USES PRESSURE VESSEL FOR STORAGE, VAPORIZER FOR FEEDING MIXER AND NH_4OH COOLER.	(11)	VAPORIZER AND COOLER NOT NECESSARY. INSTEAD OF PRESSURE VESSEL A PERMANENT EFFICIENT ''NURSE'' TANK TRUCK SPOT SHOULD BE LAYED OUT.
(12)	MOBIL INFO INDICATES CBE T/C IS TOP UNLOADING VIA TYPICAL LIQUID DIPLEG CAPABLE OF PRESSURIZING UP TO 25 PSIG.	(12)	PROVIDE 25PSIG N_2 PCV, PI AND PSV (RELIEF VALVE FOR CBE)
(13)	TO PREVENT OVERHEATING OF CBE, STEAM IS REGULATED TO 15 PSIG.	(13)	PROVIDE COMMON 15 PSIG STEAM REGULATOR FOR CBE TANK CAR COIL AND TRACING ALSO PROVIDE FOR NBE.
(14)	MOBIL PLANT SET UP FOR BOTH EDC DRUMS AND T/T'S	(14)	DEPENDING ON CONSUMPTION DRUMS COULD BE A REAL PAIN. SUGGEST TANK TRUCK UNLOAD CONNECTION.
(15)	MOBIL HNO_3 & H_2SO_4 T/T UNLOADING USES 25 PSIG NITROGEN.	(15)	PROVIDE 25PSIG N_2PCV, PI AND PSV (RELIEF VALVE)
(16)	REVIEWING ACID HAZARDS, CHECK LOCATIONS OF SAFETY SHOWERS AND EYEWASHES.		
(17)	REVIEWING SAFETY RELIEF VALVES AND RUPTURE, DECIDE BASIS FOR CALCULATION AND SIZING. SUGGEST STANDARD API STANDARD RELIEF FORMULA WITH FIRE BASIS OF LOAD.		
(18)	MOBIL SYSTEM HAS REFRIGERANT PRESS ALARM	(18)	PRODUCT 10 HAS REFRIGERANT FLOW ALARMS. DO NOT PLAN TO USE @ THIS TIME.
(19)	CHECK HYDRAULICS OF P-M101, P-M102 VS P-M107. WHAT IS POTENTIAL FOR REACTANT BACKFLOW INTO HNO_3 AND H_2SO_4 SYSTEMS? P-M107 90FT. HD OTHERS 100PSIG - SYSTEM OK.		
(20)	WATTMETER DEVICE SOUNDS GOOD FOR DETECTING AGITATOR MALFUNCTION. EXPERIENCE IS THEY ARE UNRELIABLE	(20)	USE ZERO SPEED SWITCH ONLY FOR AGITATION INTERLOCK
(21)	MOBIL INTERLOCK SETTINGS TEMP ALARM R-M101 37F TEMP ALARM AND SHUTDOWN ACID FEEDS R-M101 40F TEMP ALARM V-M101 300F		

COMMENT	REMARKS
(22) MOBIL DWGS SHOW TYPICAL PD (HNO3 & H2SO4) RELIEF VALVES BACK TO STORAGE	(22) CHECK METERING PUMPS FO INTERNAL RELIEF
(23) MOBIL SHOWS ACID SUCTION STAINERS ON HNO3 & H2SO4 PUMPS	(23) PROVIDE FOR PROTECTING OF METERING PUMP.
(24) J. FOWLER COMMENT. DO HNO3 AND H2SO4 NEED FLOW READ OUTS?	(24) PROVIDE WALLACE AND TIERNAN TYPE FLOW ROTAMETER.

CC:

(1) JOHN MILES
(2) BILL SHACKELFORD
(3) GEORGE MATHER

COPY TO BOB FABIAN

Mobil Chemical Company

CROP CHEMICALS GROUP

P.O. BOX 26683
RICHMOND, VIRGINIA 23261
TELEPHONE (804) 798-4291

December 10, 1975

REGISTERED MAIL

Mr. R. M. Fabian
Manager Business Development
Vertac, Inc.
Suite 412
2924 Knight Street
Shreveport, Louisiana 71105

Dear Mr. Fabian:

Herewith find our draft for $100,000 payable to Vertac, Inc. which covers the second and final prepayment under the provisions of the NBE toll contract.

It is my understanding that your office will take the appropriate actions to have this prepayment credited to your Eagle River Operations.

Sincerely,

D. B. Faggert
Manager, Formulations

soe

Enclosure

cc: Mr. J. W. Shackelford
Plant Manager
Eagle River Chemical Co.
P. O. Box 2648
West Helena, Arkansas 72390

Mobil Chemical Company

CROP CHEMICALS GROUP

P.O. BOX 26683
RICHMOND, VIRGINIA 23261
TELEPHONE (804) 798-4291

January 28, 1976

Mr. John Miles
Eagle River Chemical Company
P. O. Box 2648
West Helena, Arkansas 72390

AMMONIUM SULFATE WASTE

Dear John:

We have been requested to provide a certificate of analysis with each future shipment of ammonium sulfate waste. Please make arrangements to provide driver a certificate of analysis for each shipment with the following information:

Percent Ammonium Sulfate
Percent Organics
Specific Gravity

If you have any questions, please advise.

Very truly yours,

F. L. Lattire

F. L. Lattire
Manager, Product Control

soe

cc: Mr. Bruce Morrison
BFI Chemical Services, Inc.
P. O. Box 586
Theodore, Alabama 36582

CC: Lab
Shift supervisors
Mobil operators
} Please circulate tank and have analysis performed by lab prior to arrival of truck.

2/12/76

10-29-76

To: MR. JOHN O'NEILL

Mobil Chemical Company

CROP CHEMICALS GROUP

P.O. BOX 26683
RICHMOND, VIRGINIA 23261
TELEPHONE (804) 798-4291

March 25, 1976

Mr. R. M. Fabian
Business Development Manager
Vertac Incorporated
5100 Poplar Avenue, Suite 2414
Memphis, Tennessee 38137

Dear Mr. Fabian:

Based on our discussions in the meeting at Eagle River on March 23, 1976 with Mr. Shackelford and John Miles present, the following raw material usages were mutually agreed upon:

Raw Material	Established Usage * lb./lb. product	Usage, Run to Date lb./lb. product
CBE	0.840	0.835
EDC	0.125	0.158
Sulfuric Acid	0.880	0.870
Nitric Acid	0.36	0.345
Ammonia	0.055	0.053

It is understood that these agreed upon usage rates are probably upper limits and hopefully, will be improved upon as plant personnel continue to refine the process.

It is also my understanding that the amount of ammonium sulfate solution generated will be reviewed in hopes that this quantity can be reduced.

Your continued cooperation and efforts to improve in the above-mentioned areas is recognized and valued.

Very truly yours,

D. B. Faggart
Manager, Custom Manufacture

*Based on 93% CBE to 83% NBE.

soe
cc: Mr. J. W. Shackelford, Eagle River
Mr. J. H. Miles, Eagle River
Mr. R. A. Guidi, Memphis
Mr. F. L. Lattire

Mobil Chemical Company

CROP CHEMICALS GROUP

P.O. BOX 26683
RICHMOND, VIRGINIA 23261
TELEPHONE (804) 798-4291

July 29, 1975

all confidential

Copy to
John Wiles
Larry C
Jim Fowler
Bill S
Return to JFF

Mr. George Mather
Eagle River Chemical Company
Highway 242
West Helena, Arkansas

Dear George:

Enclosed you will find the following items -

1. Process flow diagrams for NBE
2. P & I diagrams for NBE
3. Standard operating conditions for NBE
4. Standard operating procedures for NBE which contain safety data sheets, physical properties of raw materials, and process description as well as specific procedures for NBE manufacture.

The analytical data will be forwarded to you from our plant chemist in Mt. Pleasant, Tennessee.

I trust this data will be handled in accordance with our secrecy agreement dated February 28, 1975.

Please advise if I can provide any additional data.

Very truly yours

D. B. Faggert
Manager, Formulations

DBF/bf
Enclosures

Copy to Beaver
John
George
Larry

Mobil Chemical Company

RESEARCH AND DEVELOPMENT LABORATORI
Dr. T. Ellison
P.O. BOX 240
EDISON, NEW JERSEY 08817
(201) 321-6249

October 31, 1977

Mr. John J. O'Neill, President
Vertrac Consolidated
Suite 2414
Memphis, Tenn. 38137

Dear Mr. O'Neill:

The Phosphorus Division of Mobil Chemical Company has asked that I send our latest toxicity information on CBE (chlorinated benzoate ester) and NBE (nitrated methyl chlorobenzoate ester).

Toxicity Test	CBE	NBE
Acute oral LD_{50} (rat)	1.00 ± 0.96 g/kg (Moderately Toxic)	4.80 ± 0.482 g/kg (Slightly Toxic)
Acute Dermal LD_{50} (rabbit)	> 5.0 g/kg (Non-toxic)	> 5.0 g/kg (Non-toxic)
Inhalation (single dose, 1 hour exp.	> 22.58 mg/l (Non-toxic)	> 21.11 mg/l (Non-toxic)
Eye irritation	Non-irritating	Non-irritating
Primary dermal	1.58 (slight irritation)	2.29 (moderate irritation)
Mutagenic (Ames)	Negative	Negative

Copies of the toxicology reports are enclosed.

If you have any questions regarding these results, please call me directly.

Sincerely,

Mobil Chemical Company
Theodore Ellison

Theodore Ellison, Ph.D.
Senior Toxicologist

TE/st
CC: W. Shackleford wo/attach.
W. Smithey, Jr. wo/attach.

Mobil Chemical Company

CROP CHEMICALS GROUP

P.O. BOX 26683
RICHMOND, VIRGINIA 23261
TELEPHONE (804) 798-4291

February 6, 1978

Mr. John Miles
Eagle River Chemical Corporation
P. O. Box 2648
W. Helena, Arkansas 72390

Dear John:

SUBJECT: EDC STUDY

The attached information was received from PPG as result of a study by National Cancer Institute. It is doubtful that method of exposure used by National Cancer Institute would be encountered in workplace, but consider it prudent that users of EDC be aware of potential damage to employees exposed to EDC.

Sincerely,

F. L. Lattire

F. L. Lattire
Manager Product Control

cbm

attachment

The National Cancer Institute (NCI) communicated to other governmental agencies and to industry trade associations the results of their rat and mouse stomach tube (gavage) bioassay screening program for ethylene dichloride (EDC). Cancer was indicated in males and females of both species. PPG has received verbal information from the MCA that rats and mice in the NCI study administered daily dosages of EDC by gavage at the maximum tolerated dose and one-half the maximum tolerated dose over the animals' life spans resulted in an increased incidence in tumors in both species of animals.

The route of administration used by NCI is not the route encountered in the workplace. Further, the dosages given to the test animals resulted in early mortality from the toxicity of EDC upon repeated treatment.

Separately the U.S. producers of EDC are co-sponsoring research in Professor C. Maltoni's laboratory in Italy. These animal studies were started in May 1976 and exposures are by inhalation, which is the principal route of exposure in the workplace. The U.S. co-sponsors have not received any information from Professor Maltoni of an increased incidence of cancer; however, this study is not complete.

The NCI studies raise the question of potential human carcinogenicity of EDC; therefore, it is recommended that all users review their operating procedures to identify inherent exposures and implement programs to contain EDC emissions to the workplace environment.

The NIOSH criteria, issued in March 1976 for EDC, recommended exposures not exceed 5 ppm as a time-weighted average. The NCI bioassay results may result in a reevaluation of the NIOSH recommended standard for EDC.

Floyd Potter

210-010-078

PPG INDUSTRIES

PPG INDUSTRIES, INC./359 NORTHWESTERN BANK BUILDING/CHARLOTTE, NORTH CAROLINA 28202/AREA 704/377-7631

THOMAS C. HARDWICK, Regional Sales Manager
Chemical Division

January 26, 1978

Mr. R. L. Warren
Purchasing Agent
Mobil Chemical Company
Industrial Chemicals Div.
P. O. Box 26683
Richmond, Virginia 23261

Gentlemen:

The National Cancer Institute (NCI) has revealed results of a long-term animal oral gavage study which showed cancer in rats and mice after EDC had been orally administered daily throughout their lives. The details of these findings, as PPG understands them, and our considered opinion, are conveyed in the attached document. We recognize that the method of administration used by NCI is not the one encountered in the workplace, but consider it prudent that users of EDC inform their workers potentially exposed to EDC and take action to identify and rectify potential exposures.

Yours very truly,

Thomas C. Hardwick
Regional Sales Manager

TCH:js

Attach.

JWS

Mobil Oil Corporation

P.O BOX 1026
PRINCETON, NEW JERSEY 08540
TELEPHONE (609) 737-3000 X2816
Dr. T. Ellison
ENVIRONMENTAL AFFAIRS
AND TOXICOLOGY DEPARTMENT

September 21, 1978

Mr. C. P. Bomar, Jr.
Director of Corporate Development
Vertac Inc.
Suite 2414
5700 Poplar
Memphis, Tenn. 38137

Dear Mr. Bomar:

I have been asked by Dr. W. R. Smithey, Jr., of the Phosphorus Division of Mobil Chemical Company to summarize additional toxicity data on Chlorinated Benzoate Ester (CBE) that the Velsicol Chemical Corp. provided the EPA under TSCA section 8 (e).

The first report covered a 96 hour static toxicity (LC_{50}) in bluegill sunfish and rainbow trout. The LC_{50} for the bluegill was 3.0 mg/liter (3 ppm) and for the trout, 7.6 mg/liter (7.6 ppm). The no-effect level for CBE in both species was 1.8 mg/liter. Behavioral observations noted irratic swimming and slowed respiration. Velsicol makes no judgement as to whether this information constitutes a significant hazard under TSCA section 8 (e). However, at the levels obtained, CBE would be toxic to fish.

In another report, the acute toxicity of CBE to the water flea Daphnia magna, was 17.7 mg/liter (17.7 ppm). This would be considered as toxic to aquatic insects. Again, Velsicol makes no judgement as to whether this information constitutes a significent hazard.

Additional mammalian toxicity indicates that CBE has an acute oral toxicity (LD_{50}) in mice of 1318 mg/kg, which Mobil considers to be moderately toxic and is similar to the LD_{50} toxicity in the rat of 1.0 g/kg.

Mobil

I will keep you informed if additional toxicity information is obtained in the future.

Sincerely,

Mobil Oil Corporation

Theodore Ellison

Theodore Ellison, Ph.D.
Senior Toxicologist

TE/ik

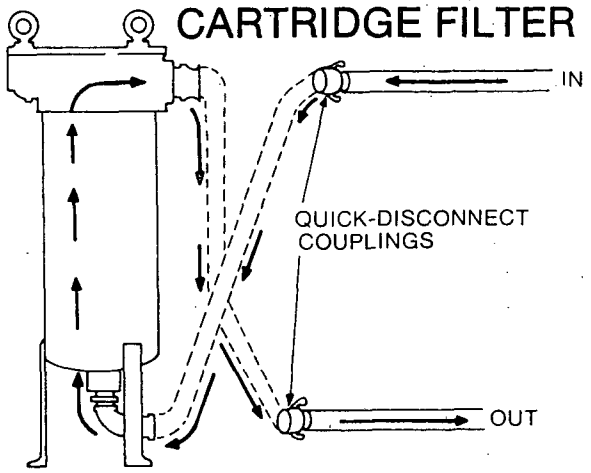
cc: W. R. Smithey, Jr.

TYPE PC FILTER HOUSING-PIPING DIAGRAMS

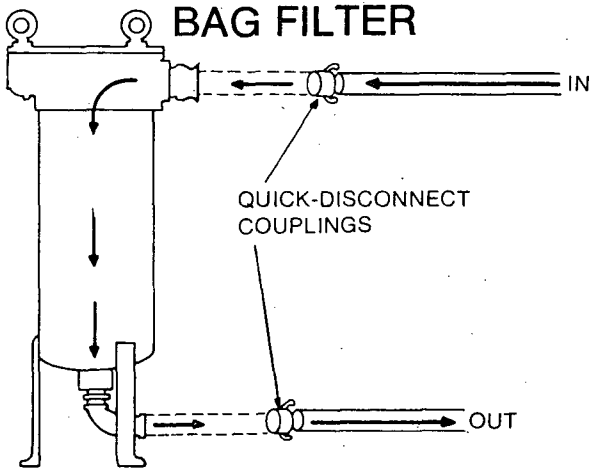
Type PC filter housings have been designed to accept either filter cartridges or filter bags. However, when changing from one to the other it will be necessary to redirect the flow through the filter. Shown below are various piping schematics when both cartridges and bags will be used in the filter housing. Locate drains for draining both filter and piping wherever convenient. Also install isolation valves.

1. QUICK DISCONNECT COUPLINGS—FLEXIBLE HOSE

CARTRIDGE FILTER

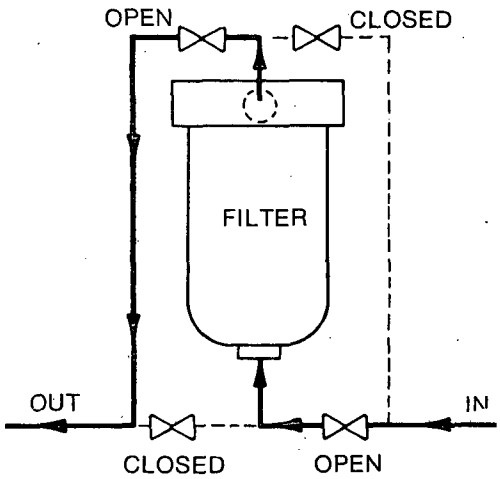


BAG FILTER

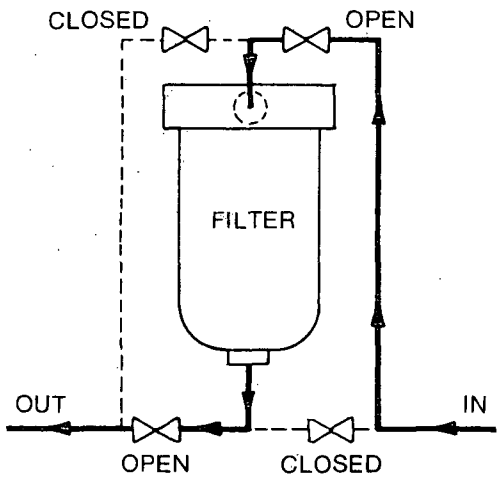


2. PIPING ARRANGEMENT WITH REGULAR VALVES

CARTRIDGE FILTER

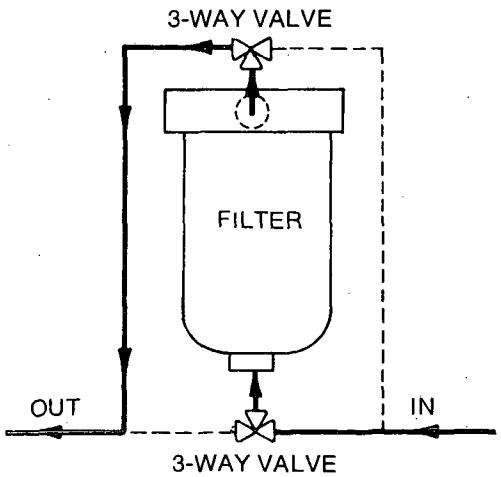


BAG FILTER

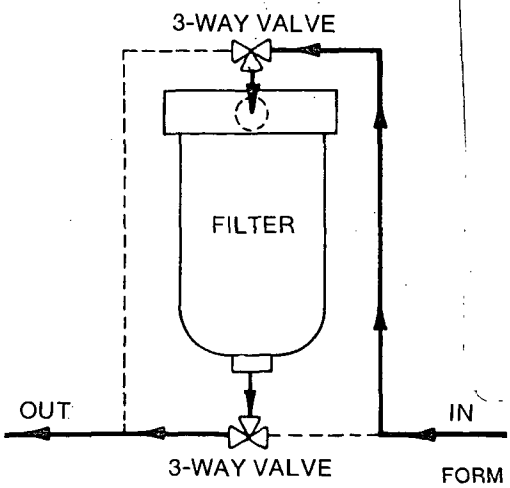


3. PIPING ARRANGEMENT WITH 3-WAY VALVES

CARTRIDGE FILTER



BAG FILTER



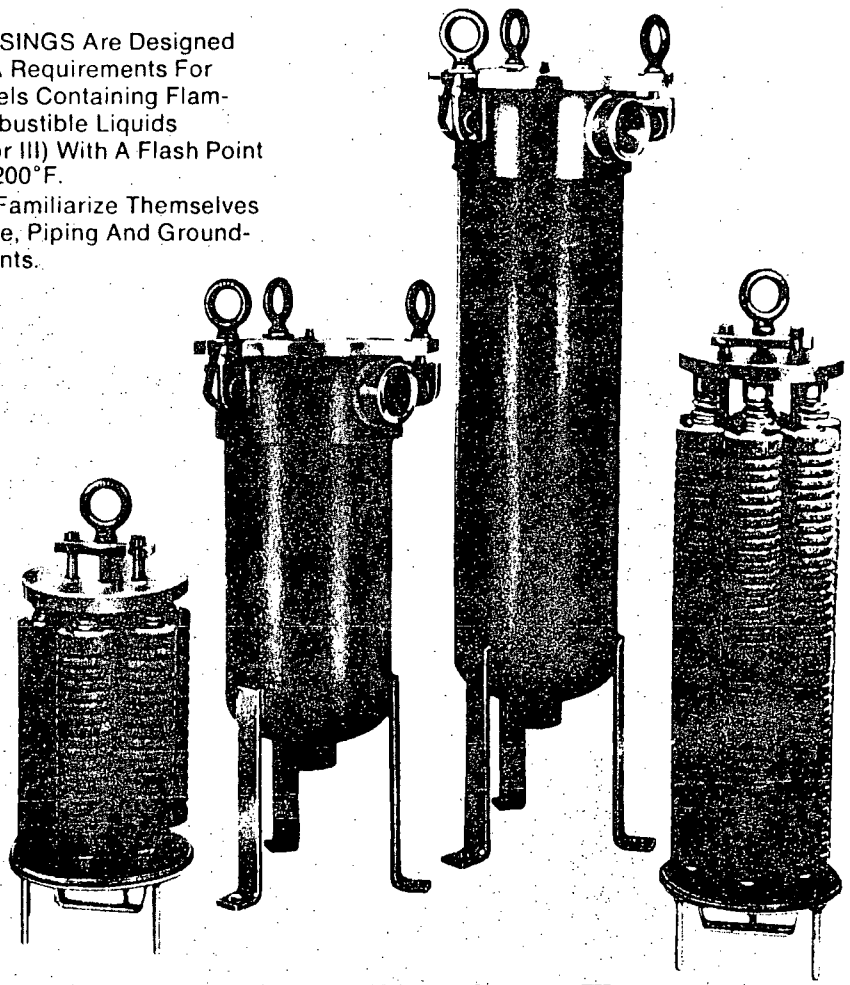
AMF | CUNO

AMF CUNO DIVISION • AMF INCORPORATED
400 Research Parkway, Meriden, Conn. 06450, U.S.A.

operating instructions

TYPE PC CARTRIDGE-PAK/BAG FILTERS

TYPE PC HOUSINGS Are Designed To Meet OSHA Requirements For Pressure Vessels Containing Flammable Or Combustible Liquids (Classes I, II, or III) With A Flash Point Of Less Than 200°F.
Users Should Familiarize Themselves With OSHA Site, Piping And Grounding Requirements.



PC Housing Design Features

- **CODE CONSTRUCTION** — housings available certified and stamped or as noncertified housings — filter vessels meet OSHA requirements.
- **BOTTOM INLET** — sludge chamber is bottom section of housing preventing sludge from contaminating the clean side of the housing, which is above the diaphragm.
- **CLEANABILITY** — no hidden chambers, crevices or internal parts . . . permits fast, thorough clean-up between color or product changes.
- **HINGED COVER** — swing bolts and hinged cover arrangement facilitates opening housing.
- **O-RING COVER GASKET** — simple, positive cover seal.
- **CHOICE OF MATERIAL** — carbon steel or 316 stainless steel — 150 psi operating pressure.

CARTRIDGE-PAK FEATURES

- **QUICK CHANGE** — entire cartridge-pak units lift vertically out in seconds — spare preassembled cartridge-pak will further minimize downtime between cartridge changes.
- **CARTRIDGE FLEXIBILITY** — cartridge-pak accommodates from 9¾" to 10" modular length cartridges. Two-high cartridge-pak can be utilized as a one-high pak when only one tier of cartridges are required. Simply install new set of one-high cartridge posts.
- **OTHER FEATURES** — includes individual cartridge pedestals, centerposts and shouldered spring and seal plates; o-ring gasket on pak diaphragm plate with sealing assured by three point contact of rods against cover.

Type PC filter housings are all-metal construction designed and manufactured to ASME code standards. Thus the housings are available as certified code vessels with ASME code Section VIII, Div. 1 inspection and National Board Number assigned; or without stamp as a noncertified code housing. Code certified housings meet OSHA requirements for pressure vessels containing flammable or combustible liquids (Classes I, II or III) with a flash point of less than 200° F.

CARTRIDGE-PAK SPECIFICATIONS

HOUSING MODEL	7PC1	7PC2
OPERATING DATA inlet-outlet maximum operating pressure flow rate, gpm	2" NPT 150 psi @ 200°F* use filter cartridge flow data in conjunction with housing flow recommendations	
CARTRIDGE SIZES	use 9¾" to 10" modular length cartridges (one or two-high configuration)	

*Housing rating only — actual filter operating temperature will be dependent upon temperature capability of the filter cartridge used in the housing.

OPERATING INSTRUCTIONS
MODEL PC CARTRIDGE-PAK FILTERS

NOTE: Filter housing, cartridge-pak hardware and cartridges are normally shipped in separate cartons. Before putting filter into service, be certain that the proper cartridge-pak assembly and correct cartridges are installed.

HOUSING INSTALLATION

Model 7PC filter housings should be installed vertically with the inlet connection at the bottom. Three mounting feet are drilled for bolting the filter in place. When locating the filter, be sure to provide clearance above the filter for removal of the cartridge-pak assembly.

Install isolation valves in lines leading to and from the filter. This will enable flow through the filter to be stopped during cartridge change-outs. Also ,install drain valves between the filter housing connections and the isolation valves so housing can be drained.

NOTE: Installation of pressure gauges in inlet and outlet piping will facilitate identifying pressure drop across the filter, thereby indicating when cartridge change-out is necessary.

FILTER CARTRIDGE INSTALLATION AND REPLACEMENT

A. Shut off inlet and outlet isolation valves Relieve pressure in housing by removing the vent plug. Open the drain valves and drain the housing.

B. Loosen the three cover eyebolts with a rod at least ⅝ inch in diameter and one foot long, being careful to back off the "hinged" eyebolt only four complete turns Swing the two free eyebolts down and lift the cover back. Grasp the lifting handle and remove the cartridge-pak assembly from the housing. Turn the cartridge-pak assembly upside down and drain the cores of the cartridges into the housing.

C. (Cartridge Replacement Only) Unscrew the piloted eyebolt on the cartridge-pak pressure plate until the spring and seal plate compression is relieved. Remove the piloted eyebolt assembly, and the spring and seal plate assemblies. The expended cartridges can now be removed by sliding them off the posts and discarded. Cartridge posts themselves may be removed by pulling them upward. The cartridge-pak assembly parts, as well as the housing, should be cleaned as necessary.

D. Position the cartridge posts by inserting them into the cartridge pedestals and pushing down until the ring near the bottom of the post is seated within the cartridge pedestal. Slide new cartridges gently over cartridge posts. **Do not force, jam or drop them in place.**

E. Place a spring and seal plate assembly atop each cartridge such that the tubular guide is within the cartridge core. Place retaining plate over spring and seal assemblies. Engage piloted eyebolt and tie rod plate end on the tie rods. Turn eyebolt clockwise to compress the retaining plate and springs until the eyebolt reaches the stop.

F. Be sure that the cartridge-pak gasket is correctly positioned and insert the cartridge-pak assembly into the housing. Make certain that the cover gasket is in place. Close cover and tighten the three eyebolts. Replace the vent plug in the cover, close drain valves and proceed to **"Starting Operation".**

STARTING OPERATION

With the vent plug removed, open the inlet isolation valve slowly and fill the filter. When liquid comes through the vent, close the inlet isolation valve, and insert the vent plug. Open the inlet isolation valve fully, and check for leaks. Open the outlet isolation valve slowly until it is fully open. Filter is now in operation.

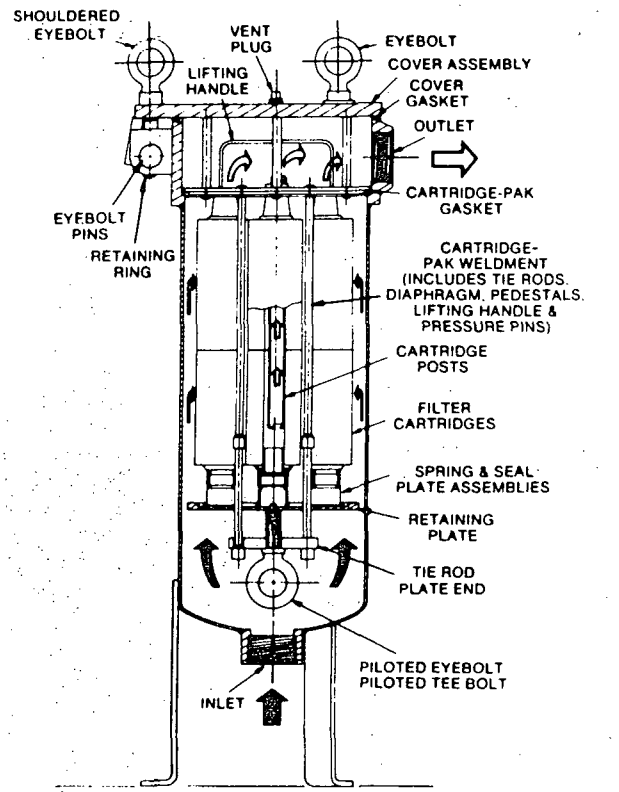
If filter leaks, close inlet isolation valve, remove vent plug to relieve pressure, and tighten cover eyebolts. **Do not overtighten.** Ideally, the eyebolts should be just tight enough to prevent leakage at operating pressure. (Max. torque value: 60 ft.-lbs.)

CARTRIDGE LIFE

AMF Cuno filter cartridges have been designed to filter a wide range of industrial fluids. The period at which cartridges should be replaced can only be determined by actual experience in your own system. However, it is generally recommended that cartridges be replaced when differential pressure across the filter reaches 25-35 psi or once per year, whichever condition occurs first.

CARTRIDGE IDENTIFICATION

The correct cartridge identification is given on a pressure-sensitive label supplied with filter cartridges. When cartridges are installed, the cartridge label should be applied to the filter label, and that exact number used for cartridge reorder.



REPLACEMENT PARTS

Your AMF Cuno Master Distributor stocks replacement parts and cartridges for your convenience. To order, specify filter catalog number, description, of part, material, and quantity required.

REPLACEMENT PART IDENTIFICATION

For Models 7PC1 and 7PC2

Assembly Numbers 44123-01 and 44123-02

DESCRIPTION OF PART	PART MATERIAL	MODEL 7PC1	MODEL 7PC2**
cover assembly	steel 316 s.s.	64417-01 64420-01	64417-01 64420-01
shell weldment	steel 316 s.s.	63952-01 63953-01	63952-02 63952-02
cover gasket	Buna N	29178-50	29178-50
cartridge-pak gasket	Buna N	32481-63	32481-63
cartridge posts*	steel 316 s.s.	63431-01 63431-09	63431-02 63431-10
spring & plate* assemblies	steel 316 s.s.	63437-03 63437-06	63437-01 63437-05
cartridge-pak weldment	steel 316 s.s.	64423-01 64424-01	64423-02 64424-02
retaining plate	steel 316 s.s.	64425-31 64425-32	64425-31 64425-32
piloted eyebolt piloted tee-bolt	steel 316 s.s.	63534-31 63536-31	63534-31 63536-01
tie rod plate end	steel 316 s.s.	63532-31 63532-32	63532-31 63532-32
eyebolt pins	steel	34845-32	34845-32
vent plug	steel 316 s.s.	31456-32 31459-48	31456-32 31459-48
eyebolts	steel	63425-34	63425-34
shouldered eyebolt	steel	63554-31	63554-31

*Seven required per assembly.

**Model 7PC2 can be used as a one-high cartridge-pak if seven cartridges are sufficient for flow and dirt requirements. When this is done, remove the two-high cartridge posts and insert seven 63431-01 posts, for carbon steel internals.

AMF | CUNO

AMF CUNO DIVISION • AMF INCORPORATED
400 Research Parkway, Meriden, Conn. 06450, U.S.A.

operating instructions

TYPE PC CARTRIDGE-PAK/BAG FILTERS

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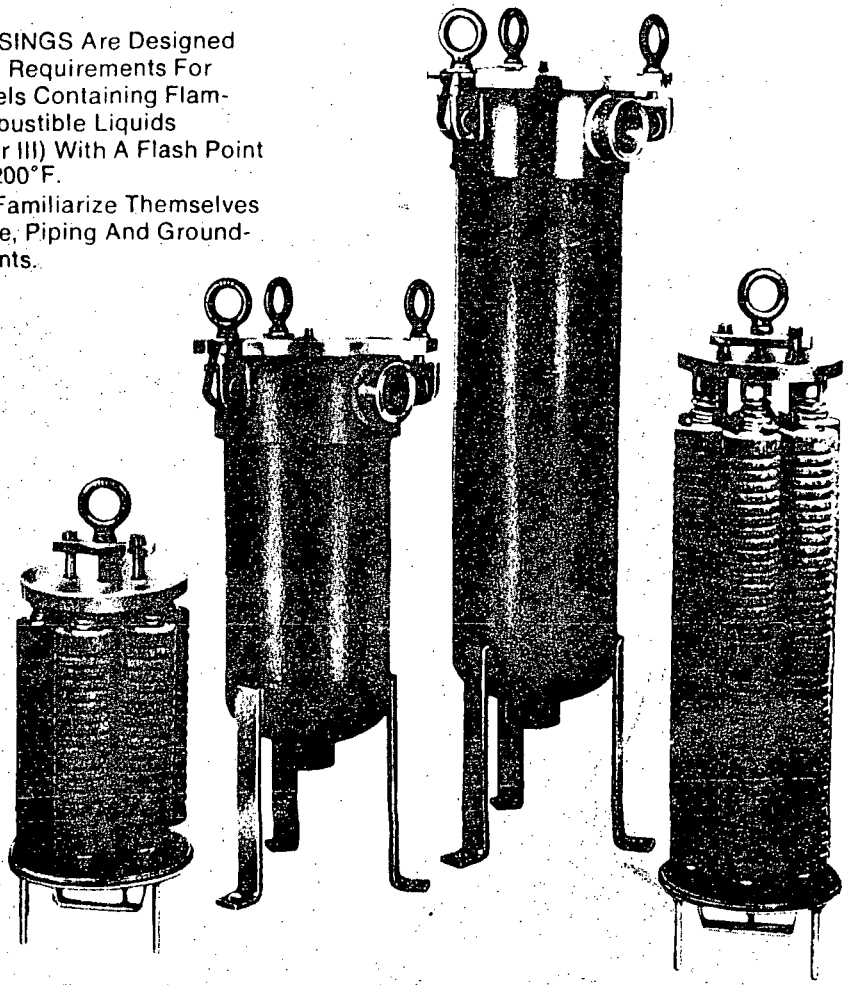

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- **CLEANABILITY** — no hidden chambers, crevices or internal parts . . . permits fast, thorough clean-up between color or product changes.
- **HINGED COVER** — swing bolts and hinged cover arrangement facilitates opening housing.
- **O-RING COVER GASKET** — simple, positive cover seal.
- **CHOICE OF MATERIAL** — carbon steel or 316 stainless steel — 150 psi operating pressure.

CARTRIDGE-PAK FEATURES

- **QUICK CHANGE** — entire cartridge-pak units lift vertically out in seconds — spare preassembled cartridge-pak will further minimize downtime between cartridge changes.
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- **OTHER FEATURES** — includes individual cartridge pedestals, centerposts and shouldered spring and seal plates; o-ring gasket on pak diaphragm plate with sealing assured by three point contact of rods against cover.

Type PC filter housings are all-metal construction designed and manufactured to ASME code standards. Thus the housings are available as certified code vessels with ASME code Section VIII, Div. 1 inspection and National Board Number assigned; or without stamp as a noncertified code housing. Code certified housings meet OSHA requirements for pressure vessels containing flammable or combustible liquids (Classes I, II or III) with a flash point of less than 200° F.

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AMF | CUNO

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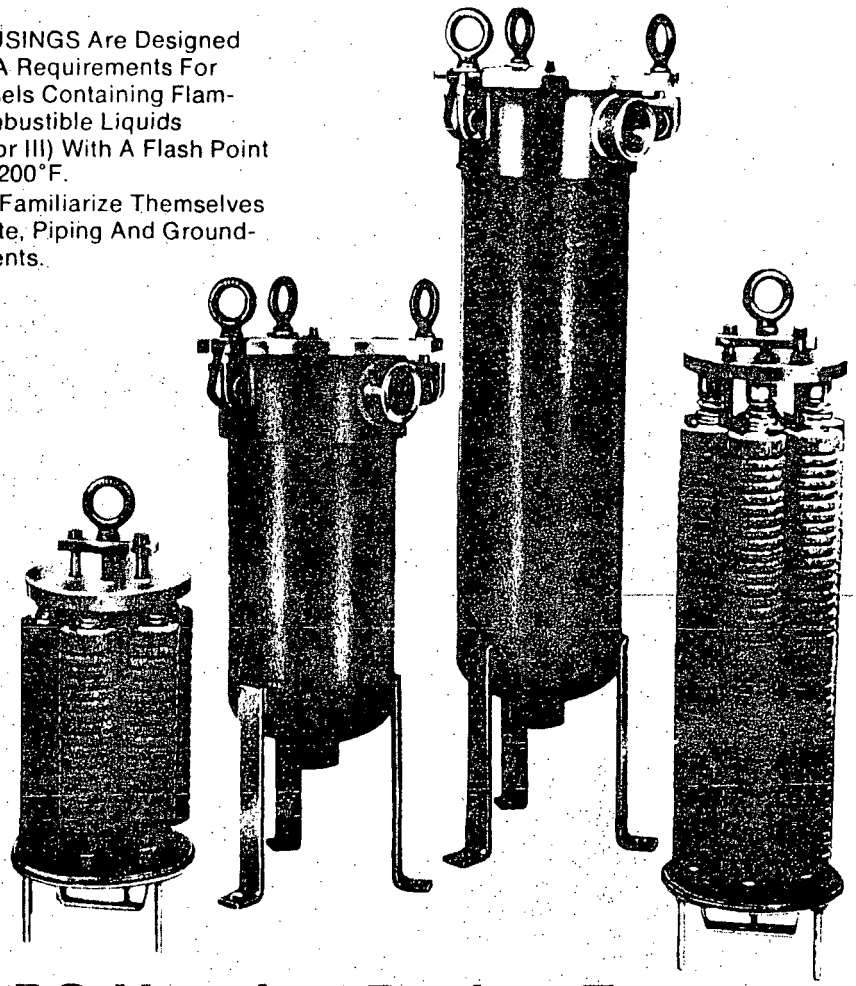

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AMF Cuno

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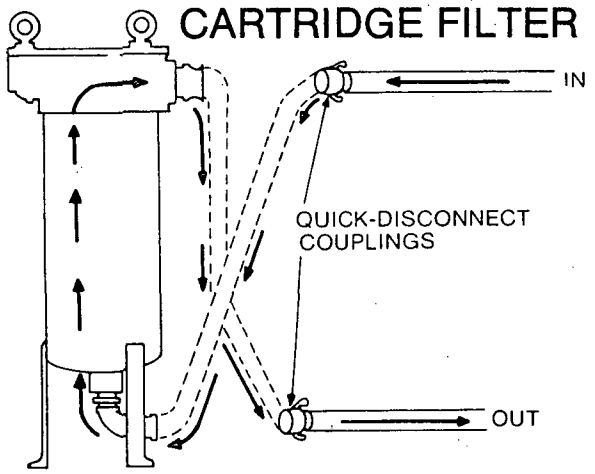
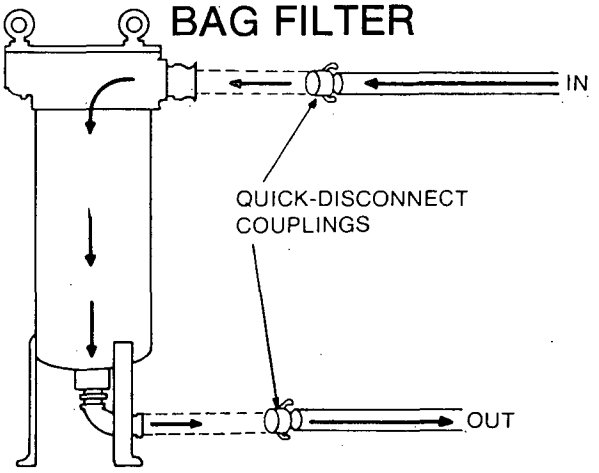
1. QUICK DISCONNECT COUPLINGS — FLEXIBLE HOSE

CARTRIDGE FILTER

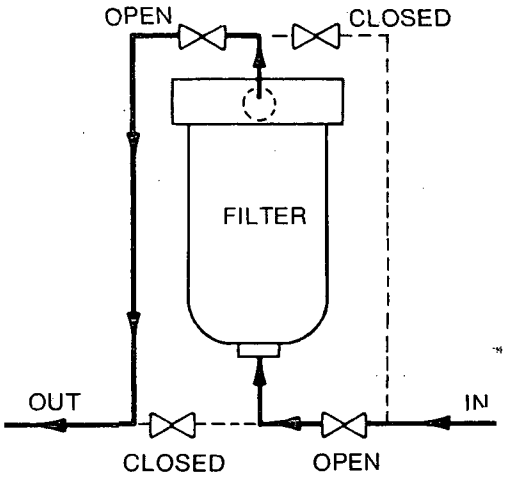


BAG FILTER

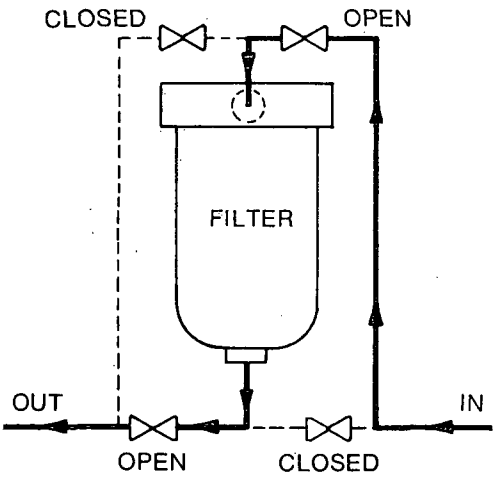


2. PIPING ARRANGEMENT WITH REGULAR VALVES

CARTRIDGE FILTER

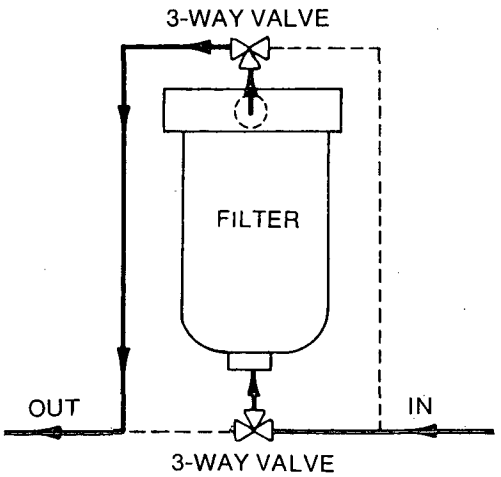


BAG FILTER



3. PIPING ARRANGEMENT WITH 3-WAY VALVES

CARTRIDGE FILTER



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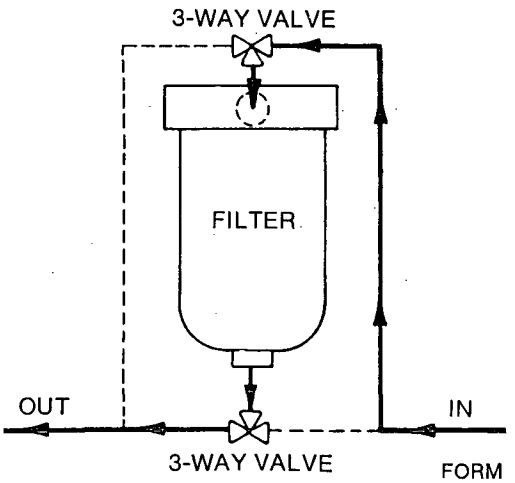

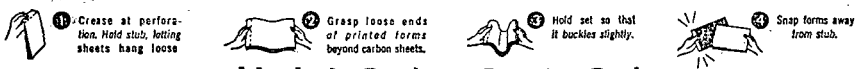

Verbal Orders Don't Go!

GrayLine "Snap-A-Way" GrayLine "Snap-A-Way" GrayLine "Snap-A-Way" GrayLine "Snap-A-Way"

SPEED MESSAGE

TO Bill Shackelford

cc: Ray Guidi

FROM **VERTAC, INC.**

MEMPHIS OFFICE

SUBJECT Mobil Nitration

Larry Graybill

DATE March 11, 19 75

Per Niven Morgan, we do not have an engineer who we can put on this one-day project. Scott Parker is in the process of moving in with his family etc. and will not be able to be at the Eagle River Plant site until the week of the 17th. Based on this, Niven suggested that we use your new engineer for this project or borrow some of George Mather's time.

I will be in touch, but it appears that we will have to let this proposal date slip to the week of March 17th.

SIGNED Larry Graybill

GrayLine "SNAP-A-WAY" FORM 44-900 2-PARTS
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SPEED MESSAGE

TO Bill Shackelford

FROM **VERTAC, INC.**
MEMPHIS OFFICE

SUBJECT Mobil Nitration Proposal

DATE April 28 19 75

Per Don Faggert, who has just returned from two weeks vacation, our proposal is still active and viable. The intermediate CBE (methyl meta-chlorobenzoate) which is supplied by Velsicol-Chattanooga has not been in ready supply, therefore, their project has been delayed by two months. Due to the delay, some of the heat has been removed from the urgency on this nitration project. Don is waiting for proposals from a couple of other sources, including Velsicol, before making a decision. However, he would expect to be in touch regarding our proposal within the next two weeks.

SIGNED

MGMMFSE MFS
2-015540E337002 12/03/75
ICS IPMBNGZ CSP
1 5015723701 MGM TDBN WEST HELENA AR 12-03 1133A EST

western union **Mailgram**

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THIS MAILGRAM WAS TRANSMITTED ELECTRONICALLY BY WESTERN UNION TO A POST OFFICE NEAR YOU FOR DELIVERY

EAGLE RIVER CHEMICAL CORP BR
PO BOX 2648
WEST HELENA AR 72390

THIS MAILGRAM IS A CONFIRMATION COPY OF THE FOLLOWING MESSAGE:

5015723701 TDBN WEST HELENA AR 36 12-03 1133A EST
FON 8047984291
DON FAGGERT

CARE MOBIL CHEMICAL
RICHMOND VA

AS OF 0700 12-3-75 WE HAVE ON HAND 47,600 POUNDS OF FINISHED NBE ON 11-28-75 WE SHIPPED 35,400 POUNDS NBE TO MOUNT PLEASANT THIS TOTAL OF 83,000 POUNDS NBE WILL SATISFY THE 12-4-75 CONTRACT COMMITMENT
JOHN MILES EAGLE RIVER CHEMICAL CORP

1136 EST

MGMMFSE MFS

REPLY BY MAILGRAM - SEE REVERSE SIDE FOR WESTERN UNION'S TOLL - FREE PHONE NUMBERS

MGMMFSB MFS
2-008377E012002 01/12/76
ICS IPMBNGZ CSP
1 5015723701 MGM TDBN WEST HELENA AR 01-12 0933A EST

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5015723701 TDBN WEST HELENA AR 31 01-12 0933A EST
FON 8047984291
DON FAGGERT

MOBILE CHEMICAL CO COPY MESSAGE
PO BOX 26683
RICHMOND VA 23261

AS OF 1-10-76 WE HAVE SHIPPED 526,000 POUNDS OF FINISHED NBE TO YOUR MT PLEASANT PLANT THIS QUANTITY WILL SATISFY DELIVERY CRITERIA PER APPENDIX B SECTION 3A OF OUR CONTRACT
JOHN MILES EAGLE RIVER CHEMICAL

0933 EST

MGMMFSB MFS

REPLY BY MAILGRAM - SEE REVERSE SIDE FOR WESTERN UNION'S TOLL - FREE PHONE NUMBERS